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PLATES TO ILLUSTRATE

MESSRS. JENKINS & RIDDICK'S PAPER

ON

"The Microscopic Examination of Metals."

JANUARY, 1905. Vol. XXX., No. 346.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, December 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. Thomas Fairley, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. P. Edgerton, J. McLeod, G. A. Pingstone, and A. W. Thorp, were read for the second time; and certificates in favour of Messrs. William Ackroyd, F.I.C., Crossley Street, Halifax, Public Analyst for the County Borough of Halifax, and John Haslam Johnston, M.Sc. (Vict.), F.I.C., Public Offices, Hampton, Middlesex, Chemist and Bacteriologist to the Hampton Urban District Council, were read for the first time.

Messrs. C. G. Moor, M.A., and W. P. Skertchly were appointed auditors of the Society's accounts for the year 1904.

The President announced that the following nominations had been made by the Council for the election of Officers and Members of Council for 1905:

President.—E. J. Bevan.

Past-Presidents (limited by the Society's Constitution to ten in number).—M. A. Adams, F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Thomas Fairley; W. W. Fisher, M.A.; Otto Hehner; Alfred Hill, M.D.; J. Muter, Ph.D.; Sir Thomas Stevenson, M.D., F.R.C.P.; J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Vice-Presidents.—L. Archbutt, B. Kitto, W. J. Dibdin.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—Alfred C. Chapman, P. A. Ellis Richards.

Other Members of Council.—Julian L. Baker, R. Bodmer, R. Hellon, Ph.D., A.R.S.M., G. T. Holloway, A.R.C.S., J. H. B. Jenkins, E. W. T. Jones, S. Rideal, D.Sc., Alfred Smetham, J. E. Stead, R. T. Thomson, L. T. Thorne, Ph.D., John White.

The President said that a special meeting of the Society would be held on January 11, 1905, when a discussion on the subject of "Brandy" would be opened by Mr. Hehner.

The following papers were read: "Some Recent Abnormal Milk Results," by Sidney Harvey; "Electric Furnaces for Laboratory Use," by Bertram Blount; and a "Note on Commercial Amyl Alcohol," by H. Droop Richmond and J. A. Goodson.

THE MICROSCOPIC EXAMINATION OF METALS.

By J. H. B. JENKINS and D. G. RIDDICK.

(*Read at the Meeting, April* 13, 1904.)

The paper deals mainly with the microscopic examination of mild steel, but the principles remain the same for all metals. In the suggestions we make respecting the preparation of the metallic sections for examination and the manipulation generally, we follow our own experience. Probably in many cases better methods exist or will be devised, and undoubtedly different metals require some modifications of treatment, according to their hardness, etc.; but we believe that the suggestions we offer will be found generally reliable and simple.

As to the size of the metal section, unless there be some special reason for examining an extensive area of the metal, it is well to keep the surface to be polished and prepared as small as possible. The work of getting a satisfactory polished surface on a large section is very great and tiresome. A piece of metal about $\frac{3}{16}$ inch thick by $\frac{1}{4}$ inch or $\frac{1}{2}$ inch square is quite large enough in ordinary cases.

Before studying wrought irons or very high carbon steels it is well to become familiar with the features of mild steel of, say, from 0·2 to 0·4 per cent. carbon content. A half-dozen re-examinations of such a piece of mild steel, watching the changes of structure which attend differences of heat treatment in a muffle furnace, will familiarize the mind with the subject better than endless description.

Those who have large numbers of specimens to examine will probably get a polishing machine, of which there are several varieties on the market; but the majority of more casual workers will be content with hand-polishing. This consists in preparing the surface with a smooth file, and then rubbing it on emery-cloths of increasing grades of fineness, finishing up with, say, Hubert's "0000" emery-paper. A final passage over some wet best quality rouge, laid on parchment or fine leather, completes the preparation. Care must be taken to avoid dust falling on the fine emery-papers and parchment. The unaided eye, after some training, can detect the development of the structure during the final stages of the polishing by reason of the harder parts standing out in relief.

It is often well to examine the polished specimens at this stage under the microscope, for some features can be seen more clearly in the polished than in the etched section. As instances of this we may mention sulphide and slag flaws in iron and steel, and cuprous oxide (a usual constituent) in commercial coppers. In the latter case the cuprous oxide can be seen located in little pits in the polished section; by vertical lighting it appears as dove-coloured dots, and as brilliant, ruby-like particles by oblique illumination. These particles of cuprous oxide can also be seen with a low-power objective in a rough *fracture* of the copper.

The etching of steel is conveniently done by dipping the polished surface into a saturated solution of picric acid in alcohol. About a half minute's immersion is generally enough, but the right time must be determined by reference to the microscope. Next to this picric acid solution, we prefer a cold, filtered infusion of liquorice root, to which is added a little recently-precipitated calcium sulphate; a piece of parchment laid on glass is moistened with this, and the polished metal surface rubbed very lightly to and fro over it. The drawback to the use of this etching agent is its tendency to decompose. Another agent sometimes useful is strong nitric acid; the polished surface of the steel or iron is touched for a moment on the surface of the acid, immediately removed, and flooded with water. The nitric acid is useful for deep etching or for picking out the grain junctions, but is not well under control.

In some cases heat-etching (heat-tinting) is useful, trusting to the difference of rate of oxidation or colour of the oxides to distinguish the constituents. Cast irons and many alloys can be conveniently etched in this way. The heat oxidation is generally effected by laying the specimen on a heated iron plate.

It must be remembered that the primary function of etching is the development of features which do not appear, or only slightly, in the unetched section. Some features are, however, best seen before etching, some with slight etching, whilst others require deep etching, and the etching agents will be selected accordingly.

In mounting the specimen it is important that the surface should be strictly parallel with the microscope stage. A simple little stepped contrivance, devised by Stead, is very useful for this purpose. A couple of them are represented near the feet of the microscope in Figs. 5 and 6. To make it, two sets of inch-wide strips of plate glass (of uniform thickness) are cut, the strips being of such decreasing lengths that when laid one on the other at opposite sides of a stout base plate of glass they form a double series of steps with a clear space between. The glass strips are fastened in position by Canada balsam. The metal specimen is placed so that its prepared surface rests on the base plate between the steps; some heat-softened cement (a mixture of equal parts of beeswax and rosin) is put on the centre of a microscope slide, and this is forthwith pressed down on to the back of the specimen until the ends of the slide rest on an opposite pair of the steps. By this means the prepared surface of the metal is mounted parallel to the microscope slide.

The specimen is now ready for examination. Next comes the question of illumination. Metals, being opaque, cannot be illuminated by transmitted light; they must be lighted from above. And here we would point out that an entirely different effect is produced according to the method of illumination. With oblique illumination one gets, as it were, the negative effect of what is got by *vertical* illumination (compare Figs. 3 and 4). This may lead to some confusion, and it is best to keep as much as possible to vertical illumination; indeed, oblique illumination is not practicable with high-power objectives.

Of methods of vertical illumination two are well known, and both depend upon the admission of a normal ray of light into the body of the microscope just above the objective, this ray of light being then reflected down on to the object which it illuminates. In the one class of illuminators (Nachet's, etc.) this reflection is brought about by a right-angled prism, fitted inside the microscope, whereas in the second (Beck's) the reflection is effected by a little plain disc of glass, which can be turned round inside the microscope tube until, at an angle of 45° with the entrant ray, the latter is reflected down on to the object.

All the vertical illuminators depending on the use of the right-angled prism are not equally effective, and we recommend that they be tested practically before they are purchased. A well-constructed one gives beautiful illumination; but, nevertheless, if it be a choice between the two types of illuminators, we recommend Beck's as being of more varied utility, and especially serviceable with high-power objectives. Beck's illuminator cannot be used satisfactorily with low-power objectives—say 1½ to 2 inch objectives. For these powers Stead has devised a simple illuminator depending on the same principle; but it is placed *below* the objective. It consists of a microscope slide, fixed or clipped in any convenient way above the metal section, and at an angle of 45°. The under surface of this slide is made to serve as a reflector, to throw a ray of light vertically down on to the section beneath.

For the photography of the metallic sections nearly every authority emphasizes the value of apochromatic objectives, but our experience with them is not satisfactory. There is a want of flatness of field with all apochromatic objectives which have come into our hands, so that generally it is impossible to get more than a minute portion of the centre of the field sharp. We prefer ordinary objectives, though they require careful selection, from whatever source they come.

In taking photomicrographs, the Welsbach gas-burner is a convenient source of illumination. If more light is desired, the limelight is very good. The electric arc-lamp, of course, reduces the time of exposure, but it is usually not so steady, and is trying to the eyes in the preliminary focussing.

We find Lumière's orthochromatic plates, series A, to give us the best results, and for printing we use "glossy" gaslight paper.

The camera arrangement is shown in Figs. 5 and 6. For the preliminary focussing the camera is swung aside (Fig. 5), so that the eye can be applied directly to the microscope. Over the eye-piece of the microscope a collarette of black cloth, with a stiff piece of cardboard behind, is seen. When, after the preliminary focussing, the camera is put back into its final position, this cloth and cardboard press lightly against the end of the camera, and form a light-tight connection. The final focussing on the ground-glass screen is done by means of a tripod lens (*vide* Fig. 5), or by an ordinary reading lens. According to the degree of the illumination and the number of diameters of magnifications, some modifications may prove useful to assist sharp focussing. Oiling the ground-glass screen, to increase its transparency, may be tried, and for very high magnifications we have found it advisable to discard the use of the ground-glass screen altogether, and to put a sheet of *clear* glass in position in the "dark slide," focussing as before by the aid of a magnifier.

Working for 150-diameter photographs, we use a ⅔-inch objective and a × 5 eye-piece. With a piece of mild steel, Lumière's plates, and Welsbach light, about three minutes' exposure is sufficient; with limelight, about 1 minute; and with arc-light, far less.

Increased stretch of camera is generally to be recommended, rather than the use of higher-power eye-pieces.

Sorby's pioneer work in the microscopic examination of steel is of the highest value, and his papers, to be found in the *Journal of the Iron and Steel Institute*, 1886, 1887, should be carefully read. Since that time the structure of steel has been studied by many workers, and the different morphological elements have received distinguishing names. For a full account, reference may be made to the successive volumes of the *Metallographist* and to the Reports of the Alloys Research Committee of the Institution of Mechanical Engineers.

It is now accepted that a simple binary metallic alloy, in cooling from a state of molten solution, acts like a solution of salt in water. On cooling the latter solution, the excess ingredient, whether it be salt or water, commences to crystallize out at a certain temperature, which depends upon the strength of the solution. This crystallization out of one constituent continues with the fall of temperature until, at a definite low temperature, which is fixed, whatever may have been the initial composition of the solution, so much of the excess ingredient has been removed that the still remaining mother liquid is saturated with respect to the *two* ingredients, water and salt. The next fall of temperature causes the whole of this remaining mother liquid to crystallize out as a minute mechanical mixture of salt and ice.

This mechanical mixture of crystals of salt and ice thus produced during the final stage of solidification is termed the *eutectic*. Its composition—the percentage of salt and ice it contains—is invariable.

It is similar in the case of simple binary metallic alloys—*e.g.*, in the case of copper-silver alloys cooling from a molten condition—the excess ingredient crystallizes out progressively until finally, at a definite, lower temperature, the mother liquid is saturated with respect to the two metals, and thereupon there is a co-precipitation of crystals of copper and silver side by side to form a mechanical mixture called the eutectic, which, of course, has a fixed composition.

If an etched section of such a copper-silver alloy be microscopically examined it will show the excess metal, whether copper or silver, which has solidified at a higher temperature, as a background, with here and there the eutectic of alternate, interstratified plates of copper and silver, which can be readily identified. This appearance of interlamination of crystal plates of the constituent metals is common to very many eutectics, but in other cases or under different conditions the mixed crystals appear more granular or confused.

In cases of other alloys—*e.g.*, zinc and copper alloys—the metals form definite chemical compounds with one another, so that we may have the case of one chemical compound dissolved in an excess of another, or separating from it on cooling. And the effects become more complicated when third metals, or compounds, are present.

In other cases, when the elements of an alloy are isomorphous in crystalline structure, the constituents may not be separately identifiable under the microscope.

In the case of steel—*i.e.*, iron-carbon alloys containing up to about 1·5 per cent. carbon—we have to consider not only the solidification of a liquid solution, but, at much lower temperatures and when the metal is solid, the separation of bodies from *solid* solution, as well as other changes. In the diagram shown in Fig. 7, in which the temperature and carbon content of the steel are co-ordinates, the temperatures at which such changes occur for different qualities of steel are shown.

The following are the points of special interest in the diagram, so far as the final microstructure of the steel is concerned. We give the views which are generally accepted. The temperatures of initial solidification of the iron are shown in the top line. Below the next line, which meets the first at 1,600° C. or thereabouts, there exists a *solid* solution of carbon in iron. At lower temperatures—say 1,000° C. to 1,100° C.—the carbon, which at higher temperatures existed as a solution in iron, now enters into a fresh condition, and although still remaining in solution, exists there now as a definite carbide of iron, Fe_3C (cementite). Disregarding here the allotropic changes in the iron, we may broadly take it that at 1,000° C., or thereabouts, we have to deal essentially with a solid solution of the carbide of iron, Fe_3C (cementite), in iron (ferrite). If the carbon in the steel is less than about 0·8 per cent.—or, we may say, rather, if the *cementite* (Fe_3C) is less than about 12 per cent.—then the iron (ferrite) is the excess ingredient; and as the temperature of the solid solution cools further, the crystals of almost pure iron (ferrite) separate out (*cf.* diagram, Fig. 7), leaving a mother solid, which becomes continuously richer in cementite, until, at a temperature of about 680° C., the mother solid is saturated with respect to the *two* constituents, ferrite and cementite, and the next fall of temperature witnesses a co-crystallization of these two constituents, generally in minute interstratified plates. The areas of the steel in which this co-separation of ferrite and cementite has taken place are known as *pearlite* areas. When examined under very high magnification, the composite nature of pearlite can be detected. Pearlite, being formed from a *solid* solution, is called an eutect*oid*, to suggest at once its relationship to eutectics, which are similarly formed from *liquid* solutions.

Under the microscope—say 100 diameters—a polished and etched section of very mild steel should show a field of ferrite, or nearly pure iron, appearing white, with here and there the almost black areas of pearlite, in which latter something of its composite character may already be indistinctly seen (*vide* Figs. 3 and 11). The total extent of the pearlite naturally becomes greater with the increasing percentage of carbon (cementite), until, when it reaches about 0·8 per cent. carbon, the whole field consists of pearlite. When the carbon exceeds 0·8 to 0·9 per cent., the *cementite* becomes the excess ingredient, and appears as the white constituent of an etched section, generally standing in relief owing to its hardness, the pearlite being black, as before.

This refers to slowly-cooled steels; but the physical properties of steel are altogether altered by the heat treatment to which it has been subjected; and this can be judged far better by the microscope than by chemical analysis, though both are essential.

For example, if a steel containing, say, 0·3 to 0·4 per cent. carbon be reheated to a temperature just short of melting, and kept there a little time, it becomes "burnt" and mechanically unreliable. In such a burnt steel one would see under the microscope minute internal cavities or fissures here and there in a section, and somewhat coarse junction markings between the enlarged grains.

If another piece of the mild steel were heated to, say, 900° C., and then quenched in water, it would be "hardened"; it would be very brittle, and not scratchable by a file. Examined under the microscope, the structure of this hardened steel will be quite abnormal, but very characteristic; the whole field will show a confused angular or criss-cross structure, called *martensite* (*vide* Figs. 31 and 36). If the same piece of hardened steel be next heated for, say, a quarter of an hour at about 350° C. and cooled, it would lose much of its hardness and brittleness, and become "tempered," and the microscopic structure will be found to have lost more or less completely the angular arrangement of martensite (*cf.* Fig. 38). If this piece of tempered steel be heated again to 350° C., and kept at that temperature for a longer time, the structure passes more completely from the martensite type, and gradually acquires the character of minute granular pearlite (*cf.* Fig. 40). The physical properties become progressively modified corresponding to these microscopic changes.

This property of steel is explained as follows: When the steel is quenched from 900° C., or thereabouts, it is cooled so quickly that the changes which would otherwise have taken place, such as the separation of ferrite and cementite to form pearlite, are prevented, and we have at ordinary temperature a metal representing more or less the condition of steel which existed at the high temperature from which it was quenched. This condition of the steel, represented in the martensite structure, is, however, unstable at low temperatures, and reheating to a comparatively low temperature—300° to 400° C.—causes a gradual reversion to a more normal condition, the reversal becoming more complete in proportion as the tempering heat is higher (up to, say, 600° C.) or the time of exposure is increased. A tempered steel has properties intermediate between those of a hardened and an ordinary steel.

Still considering such a piece of 0·3 to 0·4 per cent. carbon steel, if it were heated to, say, 1,000° C., kept for a long time at that temperature, and then allowed to cool down *very* slowly till past the "critical" temperature (about 680° C.), its structure would finally be very coarse. If this were reheated, however, for even a few minutes, to above the "critical" temperature, the coarse structure would be quite broken up, and, on cooling, a comparatively fine structure would result (*vide* Figs. 27, 32, etc.). But if the reheating of such a piece of steel were not to get above 680° C., there would be no change of structure from the previously coarse to a fine-grain condition. In this way it differs entirely from a "hardened" steel.

Whilst with steels in ordinary condition 680° C., or thereabouts, marks sharply the temperature at which the grain structure is entirely and quickly rearranged, still, if a piece of mild steel be allowed to "soak" for a very long time—say, some days or a week—at a temperature somewhat *below* 680° C., as is done in some methods of annealing, an action goes on slowly in the steel corresponding to the growth—the effect of surface tension—of the larger crystals and disappearance of the smaller, when crystals of salt stand in contact with their mother liquor. In a kindred way the crystal plates of cementite in the pearlite areas tend at a temperature somewhat below 680° C. to grow or unify, and to be represented by irregular areas of massive cementite (*cf.* Figs. 13 and 14). Any internal mechanical stresses in the steel due to "working" at too low a temperature might be relieved by such low temperature annealing, but the steel would be likely to have a somewhat low elastic limit.

If a piece of mild steel were heated for a short while to 800° to 900° C., at which temperature the cementite would go uniformly into solution, and then cooled very quickly, but not so quickly as by quenching in cold water, the rate at which the steel passes by the critical temperature, about 680° C., is still too great to allow of the satisfactory co-separation of ferrite and cementite to form well-defined pearlite. It is a condition preliminary to the formation of pearlite that the carbon at the particular point concerned shall have accumulated until it has a local richness corresponding to the eutectoid proportion, about 0·8 per cent. This is impossible in a mild steel containing, say, 0·3 to 0·4 per cent. carbon, so long as the cementite is uniformly dissolved in the mass of the metal; but during the very rapid cooling there would be an infinitude of points at which separation of ferrite (free from carbon) takes place, and from these points there would be an extrusion of the cementite so as to create a local richness in the immediately contingent metal until the carbon there reached the eutectoid proportion, and a slight separation of pearlite would follow. Further away there would be places where a condition of solution of cementite still existed, no evident separation having taken place so far as can be seen by the microscope (*cf.* Figs. 15, 25, 32).

A steel which has been cooled at such a rate as checks the separation of ferrite and pearlite, and leaves more or less of the cementite still in solution, exhibits, according to its rate of cooling, a considerable diversity of microstructure; it may touch martensite on the one hand, and ferrite with pearlite on the other. Such steel is said to be *sorbitic*, and any micro-areas in it in which there is evidence by staining that the carboniferous constituents reside, but which cannot be resolved by the microscope into pearlite, may be said to be *sorbite.*

The sorbitic condition may be attained by direct quenching in hot water or oil, or the quenching may be followed by a second process of limited exposure to a temperature of, say, 600° C.—as it were, a process of over-tempering.

This sorbitic structure is worthy of attention, for steel-makers are now devoting much attention towards developing this condition in steel intended for engineering purposes. A sorbitic steel may be regarded, perhaps, as intermediate in mechanical properties, as well as in microscopic appearance, between tempered steel and ordinary annealed steel. Its total strength is increased, but its elastic limit is increased in a still higher ratio by this treatment.

Thus, in general, the changes of structure in a steel which attend differences of heat treatment—of which many illustrations are given in the accompanying figures—are readily followed by the microscope, and since there are modifications of mechanical qualities corresponding to all these changes, it will be evident that the microscope is an invaluable aid to chemical analysis, enabling us to foretell the physical properties of any specimen of steel, or to account for a local weakness.

The value of the microscope is not so marked in respect to the other ordinary elements of steel. In the case of sulphur, however, the rôle played by manganese is seen. If manganese be absent the sulphur exists as a fusible sulphide of iron, often lying as a brittle constituent between the junctions of the crystalline grains, somewhat as shown in Fig. 8. If sufficient manganese be present, however, it seizes the sulphur and forms a less fusible sulphide, and then appears—so far as we have observed—invariably locked up in the *ferrite* areas, almost as though the particles of sulphide of manganese had served as nuclei round which the ferrite grains had crystallized. An impurity in this condition is far less likely to seriously affect the strength of the steel than when it resides at the grain junctions. Another property of the manganese in steel may be here mentioned. High manganese often, but not invariably, renders the pearlite areas larger than would correspond to the carbon content, and more or less sorbitic in character (*cf.* Fig. 15). Apart from combination as sulphide, the manganese seems to reside largely as a carbide in the pearlite areas.

Phosphorus and silicon in any quantity likely to be present in a mild steel appear to reside in the ferrite, and at present we cannot point with confidence to any definite features under the microscope to correspond with them; but it is probable that further experience will show special properties in the ferrite grains—*e.g.*, difference of action on being etched or heat-tinted—to correspond with the content and condition of these impurities.

When the phosphorus is present in large quantities, as well as much carbon, it leads to the formation of a phospho-eutectic of characteristic appearance (*vide* Fig. 3). This is a usual feature of cast or pig iron.

Of course, if the phosphorus or silicon were oxidized, it would appear as slag in the steel.

The examination of a metal or alloy is generally conducted with a view of its past history or its intended use. In case of the failure of a metal in service the examination of sections in the immediate neighbourhood of the point of failure often shows a purely local defect to which the failure is attributable; or the structure of the metal, as a whole, may be bad. A small-grain structure and an absence of linear arrangement is generally desirable, and this should be secured by proper annealing. If cooling curves can be consulted with respect to any alloy, they help in the interpretation of the microscopic appearance. It will be well to examine the sections when simply polished, and then when etched to different depths. In the case of steels, for instance, it is not often that the degree of etching which brings up the pearlite into best definition will enable the junctions between the ferrite grains to be seen; these generally require much deeper etching. It is important to examine these grain junctions with suitable (generally high) powers. Impurities in alloys and metals often lie along these grain junctions, and constitute sources of weakness in the metal. In other cases, however, these junctions are of the toughest character, and the grains will themselves split across rather than the junctions give way or open. Often it is well to get a thin disc of the metal, which is polished, and then bent in a vice or a V-block. A fresh examination of the bent surface will often show whether a metal is stronger in the grain or in the junctions.

EXPLANATION OF FIGURES.

In the photomicrographs it may be taken, unless otherwise stated, that the sections of metal are polished and etched and vertically illuminated.

Figs. 1 and 2: Iron crystals. Actual height of No. 2 crystal is 7 inches. When a metal solidifies, such crystals, starting from an infinitude of points, continue to grow until they are arrested by encountering other growing crystals. In consequence of this interruption to crystal growth, the crystalline nature of the grains of steel is rarely evident in their contours; but the internal orientation and other crystal characteristics of the grains are evident when the metal is strained. Free iron crystals, such as are illustrated, are found fringing hollows in steel castings where the crystal development has been uninterrupted.

Figs. 3 and 4: Pig-iron. Illustrates the different effect of vertical and oblique illumination of the same section. Fig. 3 is vertically illuminated. In it the graphite plates form white, almost straight lines; they are surrounded by nearly black pearlite, and then come irregular white areas of a phospho-eutectic. Fig. 4 is of the same section, but obliquely lighted. ×100 diameters.

Figs. 5 and 6 show camera and microscope. In Fig. 5 the camera is swung aside for preliminary focussing; in Fig. 6 it has been put into position.

Fig. 7: Cooling curves of iron-carbon alloys (steels).

Fig. 8: Wrought iron, practically carbonless. Has been heated almost to "burning"-point. The contours of the crystal grains are very distinct. The dark specks in this are *not* of pearlite. Some slag impurities lie along some of the junctions of the ferrite grains, and some highly minute crystal cubes of iron exist in a few of the grains. ×150 diameters.

Fig. 9: Very mild steel, 0·05 per cent. carbon. Dark pearlite areas can now be seen on a general ferrite background. (*Cf.*, Fig. 13, which shows the same steel, in which the pearlite is largely replaced by cementite.) ×150 diameters.

Fig. 10: Mild steel, 0·18 per cent. carbon. The areas of pearlite become greater in accordance with increased percentage of carbon. ×150 diameters.

Fig. 11: Mild steel, 0·4 per cent. carbon. The dual structure of the pearlite can be indistinctly seen. ×150 diameters.

Fig. 12: Cemented steel. The carbon in this is much beyond 0·8 per cent., and here we have *cementite* as the white constituent, pearlite constituting the general background. ×150 diameters.

Fig. 13: Cementite produced in very mild steel (0·05 per cent. carbon; *vide* Fig. 9) by annealing below the critical temperature. ×150 diameters.

Fig. 14: Cementite produced in mild steel, 0·17 per cent. carbon, by long annealing at about 600° C. ×300 diameters.

Figs. 15, 16, and 17 illustrate steel containing high manganese (1·66 per cent.) and sulphur (0·13 per cent.). The sulphide of manganese will be seen to reside in the ferrite. The mechanical working of the steel at a very high temperature has drawn out the sulphide into threads, as can be seen in the longitudinal section (Fig. 15), whereas in the transverse section (Fig. 17) the threads, being cut across, appear as dots. Note, too, that the extent of the pearlite is greater than would correspond to the content of carbon (0·27 per cent.). This enlarged structure is often found in steel containing high manganese. The steel is somewhat sorbitic.

Fig. 16 is of the same steel, but illustrates that the sulphide threads can be even more plainly seen in the polished section before etching. ×150 diameters.

In nearly every mild steel such threads of sulphide or slag are evident to a greater or less extent in the longitudinal sections. If the "working" of the steel in manufacture has been continued down to a low temperature (below the critical temperature), the grains of pearlite will also be drawn out, so that there is a general alignment in the section. If the steel, after such working, be again annealed, the pearlitic structure will be quite broken up at temperatures which will be far below those that are necessary to alter the features of the sulphide threads, which latter will still remain, therefore, to show the direction of "working." It is probably due to the relative permanence of position of these impurities that there is a tendency for lines of ferrite to reappear where they previously existed, although the steel has been reannealed.

Fig. 18: Enlarged view of grain of pearlite. It appears to be made up of alternate micro-plates of hard cementite and soft ferrite. Etching eats away, more or less, the soft plates of ferrite, and the nearly black appearance of etched pearlite is due to shadows cast by the projecting edges of the minute cementite plates. ×350 diameters.

Fig. 19: Oxygen in copper. The oxygen, which makers purposely leave in commercial coppers, resides in minute pits as ruby-red cuprous oxide (or, it may be, as an eutectic of cuprous oxide and copper). The consequent breaking up of the solid continuity of the copper would appear to be disadvantageous; it is said, however, to be necessary to counteract the bad effect of certain metallic impurities, such as lead or bismuth. The left-hand figures show the coppers polished only; the right-hand figures show the same after etching with nitric acid. The oxygen-content of the three coppers is respectively 0·01 per cent., 0·1 per cent. (about average quantity), and 0·33 per cent.; the latter is, undoubtedly, excessive, and the metal brittle as a consequence. ×65 diameters.

Fig. 20: Burnt copper. A "burnt" metal generally shows big grains and a coarsening of junctions. ×25 diameters. Oblique light.

Fig. 21: Small V-block of copper (0·33 per cent. O.) which was exposed for about three-quarters of an hour at somewhat low red heat to the action of hydrogen (*cf.* Archbutt's paper on determination of oxygen in copper, ANALYST, October, 1900). The block was then cut across and the section polished. The line to which the oxide has been reduced is clearly marked; the darker interior portion is still charged with the oxide, but the outside portion shows none of it under the microscope. The depth to which the action extends seems proportional to the time of exposure. ×10 diameters. Oblique light.

Fig. 22: A polished section of copper was heated as above in hydrogen. The action of the hydrogen, besides reducing the oxide, also seems to develop the grain contours; possibly the gas finds entrance to the interior of the copper largely past the grain junctions. ×150 diameters.

Fig. 23: Piece of mild steel, polished, then heated in hydrogen as above; the picking out of the grain-junctions is again apparent. ×150 diameters.

Fig. 24: Copper meshed with small percentage of bismuth. ×65 diameters.

Fig. 25: Piece of mild steel, 0·35 per cent. C, which has been specially "sorbitized" by manufacturers. The structure at some places approaches martensite, but more generally it shows a structure, as represented in Fig. 25, which is intermediate between that of Fig. 38 and that of Fig. 40. ×150 diameters.

Fig. 26: Mild steel, 0·43 per cent. C, with strongly angular structure, not properly annealed. ×150 diameters.

Fig. 27: Same steel as Fig. 26 after annealing. ×150 diameters.

Figs. 28 and 29: A mild steel shaft (0·19 per cent. C) showed a minute "hair-line" flaw on surface. It was condemned, and broken across at the place of the flaw. The fracture showed at one point of its circumference a projecting lip (B, Fig. 28). It was cut through along the line A B (Fig. 28) so as to show the lip in profile. This section, when polished and etched, showed that, whereas the direction of grain in the main body of steel was longitudinal, as indicated by the arrows, yet near the lip there is a distinct sweeping back of the grain into the lip. The explanation would appear to be as follows: When the red-hot shaft was being worked under the hammer it was subjected to a process by which its diameter was reduced. In the course of this reduction a portion of the metal got puckered up, then folded over and hammered down flush. The temperature of the metal at this time was too low to let this folded-over portion weld with the mass of metal beneath, so it remained as an "overlap."

Figs. 30 and 31: Fig. 30 shows the section through the chimb-joint of an electrically-welded drum. It is made up of four thicknesses of mildest steel the flush edges of which have been fused together by the momentary impinging of an electric arc. The metal at the edge was thereby instantly raised to boiling temperature of steel, from which the body of cold metal chilled it with almost as great rapidity. The line to which fusion has extended is clearly seen.

Fig. 31 shows the structure at the line to which the fusion extended; the upper part shows a martensite structure, whereas in the lower part the original structure of normal very mild steel remains unaltered. The line of separation of the two structures is very sharp. The diameter of the disc of metal represented in the photomicrograph (Fig. 31) is less than a millimetre. ×65 diameters.

Fig. 32: Illustrates the changes of structure produced by simple heat treatment in a piece of mild steel (0·35 per cent. C). ×150 diameters.

Fig. 33: Cast-steel bar (0·32 per cent. C and 0·34 per cent. Si). This imperfect bar was tested to destruction, and broke at two places a few inches apart. The fractures thus exposed were entirely different, the one being very fine-grained (AA, the outside figure), whilst the other was coarsely crystalline (BB, outside figure). The piece was then sectioned from the one fracture to the other, and the section polished and etched. The centre figure shows the etched section full size. It will be seen that the transition from the fine to the coarse structure is fairly sharply located between "3" and "4." In the lower figure are photomicrographs of the steel, ×25 diameters, taken at the points 1, 2 . . . 8. It is manifest that if the two fractures had taken place within ½ inch of each other at 3 and 4, the one fracture would still have been very fine and the other very coarse. The explanation of the difference of structure is as follows: the main portion of the bar, represented by BB fracture, had not been annealed to any sensible extent, and hence its coarse structure. The other, AA, end of the bar had been reheated for further working. In this reheating the temperature to the *left* side of 3 had risen above the "critical" point, and a breaking up of the original coarse structure had followed; but on the right side of 4 the temperature had not reached the "critical" point, and the coarse structure remained. Figs. 34 to 41 will show variations of structure which have attended further heat treatment of this steel.

Figs. 34 to 41: Several strips of the steel shown in Fig. 33 were taken, as represented between Figs. 1 and 8, and they were subjected to different heat treatments. Fig. 34 shows the fine structure, whilst Fig. 35 shows the coarse structure, corresponding respectively to 1 and 5 of Fig. 33. One of the strips was heated to very high temperature, almost "burning," and then quenched in water. At each part of the strip we had the structure (martensite) of "hardened" steel (Fig. 36); it could not be "touched" with a file. This hardened strip was next heated at 350° C. for a quarter of an hour; it was thereby "tempered," and lost much of its hardness. The structure was now very confused, but still showed traces of martensite (Fig. 38). It was heated again to 350° C. for six hours, this continued the tempering process, and the steel now had the structure represented in Fig. 40. It is difficult to give a name to this structure which is not open to objection; by some it is called minute granular pearlite. When this same piece of steel is heated up well above the critical temperature, and allowed to cool somewhat quickly, it gets the structure of Fig. 41—not very different to Fig. 34.

A portion of the coarse end of a strip of the steel was heated up to 350° C. for six hours. It showed no alteration of structure (*cf.* Figs. 35 and 37). It is not, of course, the same actual section shown in the two views. It had to be repolished after the heating.

Another strip of the original steel was taken, heated up to about 850° C., or thereabouts, for a few minutes, and then rapidly cooled between two iron blocks. All parts of the steel had the very fine-grain structure represented in Fig. 39.

It must be understood that small pieces of metal can be heated and cooled rapidly with comparative ease; but manufacturers dealing with large and irregular masses have many practical difficulties to deal with, and allowance must be made for this. What one would desire is not always what we can fairly expect in the way of structure in large masses of steel. But quite apart from the question of chemical purity, there is a general recognition now on the part of the best makers of the value of a fine-grain microscopic structure to secure the best properties in steel.

DISCUSSION.

The PRESIDENT (Mr. Fairley) said that with regard to this subject he was very much in the position of a learner. Among those present, however, whose work was specially well known in connection with it was Professor Huntington, whom he would ask to be good enough to open the discussion.

Professor HUNTINGTON said that, as a matter of fact, all who had to deal with metallurgy were very much in the position of learners. There was an enormous amount of learning for them to do. He thought there could be no doubt that the microscopical examination of metals was indispensable if their structure and the treatment they had undergone were to be fully understood, and the paper which had just been read would be very valuable as an introduction to the subject for those who had not already gone into it, while one could not possibly get any harm in listening to such a paper as this even if one had studied the subject. The photographs were most admirable and interesting. In the case of such a paper it would not be fair to make any carping criticism—in fact, the paper was not open to it. The subject, however, was open to any amount of discussion. The difficulty was that one did not know where to begin or where one would end, and at the late hour which had been reached one hardly liked to begin. With regard to arc-lamps, these had been used in his own laboratory for some time with satisfactory results. He had begun by using oil, and from that had gone on to the use of the arc, so that he could not speak much about the oxyhydrogen light. The arc, however, was very useful if the necessary electric current was available. It was simple to handle and very effective, and the lamps were comparatively cheap. A good arc-lamp could be bought for about £5. The difficulty was not so much with the lamps as with the carbons. The whole secret rested in the selection of suitable carbons free from ash. Bad carbons, owing to the ash which they contained, caused the light to flicker considerably, which at once destroyed the clearness of the photographs. He had obtained very satisfactory carbons from Messrs. Siemens. Many people seemed to think that it was necessary to have some automatic arrangement, but he did not find that at all necessary. He had only a hand arc-lamp, but the time of exposure was really so short that it was quite easy to regulate the arc, the consumption of carbon not being so rapid as to cause any difficulty. In fact, although he had been quite prepared to get an automatic lamp if it were really found to be necessary, he had not yet done so. With regard to copper, Heyn had done a great deal of work, having been perhaps the first to investigate the eutectic of oxide of copper and copper; and there appeared every probability of an easy method being afforded in this way of actually estimating the oxygen present in otherwise pure copper. There was an actual eutectic, which was quite distinct, and it was perfectly easy to estimate practically by the eye, without going into fine measurements of areas, the amount of oxygen present. He believed (but he spoke subject to correction) that that method was actually being used at Woolwich at the present time as a quantitative means of estimating the amount of oxygen in copper, and in his own laboratory, without going very far into the subject, photographs had been taken connected with actual practical work, and it had been found possible to obtain a very close approximation to the amount of oxygen present. It could be done roughly by simply comparing the photographs taken with Heyn's diagrams—a number of photographs showing the effect of different amounts of oxygen. Some five-and-twenty years ago he had worked with Professor Guthrie on the subject of the cryohydrates, as they were called by Professor Guthrie, who did not then use the term "eutectic." Professor Guthrie had thought these to be definite chemical compounds—an idea which he (the speaker) must say had not appealed to his mind at the time, as it seemed strange that any chemical compound should exist having hundreds of molecules of water. Later on Professor Guthrie went on to apply the same idea to alloys, and it was then that he originated the term "eutectic," which he applied to the lowest melting alloy in a combination of metals. Of course, the authors were quite right in comparing the two things, though Professor Guthrie originally did not use the term "eutectic" at all in connection with the cryohydrates.

Mr. Blount said that it would not be wise at that time of the evening to speak at any length, but he felt—and he thought he might voice the congratulations of others also—that the only word that would properly describe the work that the authors had done, and the illustrations that they had given of it, was "splendid."

The President expressed much pleasure in supporting what Mr. Blount had said. The Society was exceedingly indebted to the authors for the very lucid and interesting account they had given of this subject. There were many points that one would like to talk about, but that must be reserved for a future occasion.

Mr. Archbutt said that he had brought two specimens which might perhaps be interesting, bearing upon the photographs which the authors had shown. In pointing to the effect of oxygen in copper, Mr. Jenkins had remarked that, although it was undoubtedly useful, it was, perhaps, rather a pity that it should be there. One of the specimens which he (Mr. Archbutt) had brought had been made by melting electrolytic copper with 0·15 per cent. of lead. An attempt had been made to forge it at a red heat, but it was practically unforgeable. The second specimen was a piece of copper containing the same percentage of lead as the first, but with the addition of 0·12 per cent. of oxygen, added in the form of cupric oxide, and this piece had forged perfectly well. He should like to ask Professor Huntington whether the method of estimating oxygen by the microscope was applicable to copper which had been mechanically treated, because cast copper containing oxygen presented a very different appearance from that of copper which had been rolled, drawn, or hammered. In cast copper the oxide formed a mesh throughout the copper, whereas in copper that had been drawn, rolled, or hammered the oxide was distributed in the form of dots and bands, as shown in the authors' photographs. In the case of the copper to which lead had been added, but no oxygen, the lead was seen on microscopical examination before hammering to be distributed throughout the crystalline grains in particles, which in some way or other caused the effect he had mentioned; whereas in the copper which contained oxygen as well as the lead, the whole of the lead had been drawn into the oxide network, which was presumably the reason why it was not deleterious in that piece of copper, while it was deleterious in the other piece in which it was distributed throughout the grains. He would like to add his hearty congratulations to the authors on having presented so interesting a paper and such excellent photographs.

Professor HUNTINGTON said that Mr. Archbutt was no doubt correct in saying that there was a considerable difference in the appearance as regards the distribution of the oxygen between cast copper and wrought copper. It was not, however, a point that he had worked at much himself, though he had made some observations on it. A great deal of work required to be done on the oxygen question, but he had not had time to do very much himself. Indeed, it would probably take many months if one started on it at all.

Mr. ROSS said that in the apparatus used in a sulphate of ammonia manufactory, the old saturator that had been in use for about twenty years wore very much better than a newer one made of much purer lead. The new one corroded at the seams, though the lead used for the seams and for the body was from exactly the same mass. Speaking from recollection, he thought there was not very much difference in the two cases between the amounts of oxygen which the lead contained, but possibly microscopical examination would have revealed some difference in the structure.

Mr. JENKINS, in reply, said that it was evident that by examining a polished section of copper under the microscope a fairly good estimate of the percentage of oxygen could generally be made. The chief difficulty was that the oxide was not evenly distributed in the metal sections. But whatever observations were made under the microscope, it would be necessary finally to compare them with determinations of oxygen made by some standard chemical method, such as ignition in hydrogen, as recommended by Mr. Archbutt. Mr. Riddick and he were grateful to the meeting for the attention with which the paper had been received. He desired to express his own indebtedness to Mr. Riddick, who was very largely responsible for the execution of the photographic work, and had brought a great deal of skill and patience to bear upon it.

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REPORT OF THE COMMITTEE APPOINTED TO CONSIDER THE STANDARDIZATION OF METHODS FOR THE BACTERIOSCOPIC EXAMINATION OF WATER.

(*Reprinted from "The Journal of State Medicine," August*, 1904.)

At the Congress of The Royal Institute of Public Health, held in the University of Liverpool, in July, 1903, a resolution was carried, on the motion of G. Leslie Eastes, Esq., M.B., B.Sc., that it was desirable that a committee should be appointed to consider the methods employed in the bacterioscopic analysis of water, and, if possible, to draw up a scheme of uniform procedure for adoption in such examination, and to report to the next Congress of The Institute.

The Council of The Royal Institute of Public Health, in accordance with this resolution, appointed a committee of the following gentlemen:

Professor Rubert Boyce, M.B., F.R.S. (chairman); the President of The Royal Institute of Public Health, Professor William R. Smith, M.D., D.Sc., F.R.S. Ed.; George M. Duncan, Esq., M.D.; G. Leslie Eastes, Esq., M.B., B.Sc.; John Eyre, Esq., M.D.; Major W. H. Horrocks, R.A.M.C., M.B., B.Sc.; William G. Savage, Esq., M.D., B.Sc.; Professor G. J. McWeeney, M.D.; Professor G. Sims Woodhead, M.D., F.R.S. Ed.; and Professor R. Tanner Hewlett, M.D., D.P.H. (Honorary Secretary).

REPORT OF THE COMMITTEE.

All the members of the committee are in agreement that the minimal number of procedures should be:

(*a*) Enumeration of the bacteria present on a medium incubated at room temperature (18-22° C.).

(*b*) Search for *Bacillus coli*, and identification and enumeration of this organism if present.

The committee regard these procedures as an irreducible minimum in the bacterioscopic analysis of water. The majority of the committee recommend in addition:

(*c*) Enumeration of the bacteria present on a medium incubated at blood heat (36-38° C.).

(*d*) Search for and enumeration of streptococci.

The committee do not think it necessary as a *routine* measure to search for the *Bacillus enteritidis sporogenes*, but are agreed that in special or exceptional instances it may be advisable to look for this organism.

THE COLLECTION OF THE SAMPLE.

No special precautions beyond those generally recognised are suggested for taking the sample. The samples should be collected in sterile stoppered glass bottles having a minimal capacity of 60 c.c. In special instances it may be desirable to have much larger quantities.

Unless examined within *three* hours of collection the sample must be ice-packed. (The committee recognise that under all circumstances the sooner the water s examined after collection the more reliable are the results obtained.)

MEDIA TO BE EMPLOYED FOR ENUMERATION.

The choice of medium lies between distilled-water gelatin, nutrient gelatin, distilled-water agar, gelatin agar, and nutrient agar. The reaction of the medium is of importance.

For enumeration at room temperature any of these media may be employed, but for enumeration at blood heat an agar or gelatin agar must be used.

The Americans seem to be using an agar medium only, and although on the ground of simplicity it might be desirable to use a single medium for enumeration under all circumstances—*e.g.*, a distilled-water agar—it is felt by the committee that gelatin media frequently give indications of value that are lacking with agar—viz., liquefaction of the medium by many organisms and the more characteristic appearance of the colonies in it; gelatin is therefore recommended.

Since with a polluted water (detection of pollution being the ultimate aim in water examination) nutrient gelatin gives a relatively larger number of colonies than distilled-water gelatin, nutrient gelatin should be used when one gelatin only is employed. At the same time, it is recognised that cultures in distilled-water gelatin compared with cultures in nutrient gelatin often give useful indications. Thus with an unpolluted water the number of colonies is usually relatively larger in distilled-water gelatin than in nutrient gelatin; with a polluted water the converse is the case. Therefore the use of *both* gelatins (distilled-water and nutrient) is desirable, sets of plates being made with each medium.

Similarly, it was felt by many members of the committee that a comparison of the ratio of the number of organisms developing at room temperature to those developing at blood heat gives useful indications. With a pure water this ratio is generally considerably higher than 10 to 1, with a polluted water this ratio is approached, and frequently becomes 10 to 2, 10 to 3, or even less. The actual number of organisms growing at blood heat is also of considerable value apart from any question of ratio. Therefore it is suggested that plates of nutrient agar should also be employed and incubated at blood heat.

In certain instances it is true that this ratio may be unreliable. Thus with surface waters, especially in tropical countries (as pointed out by Major Horrocks), varieties of the *Bacillus fluorescens liquefaciens* and *non-liquefaciens* and *Bacillus liquefaciens* may be abundant, and grow well at blood heat.

PREPARATION AND REACTION OF MEDIA FOR ENUMERATION.

(*a*) *Distilled-Water Gelatin.*—Ten per cent. gelatin in distilled water, and brought to a reaction of + 10 (Eyre's scale).

(*b*) *Nutrient Gelatin.*—Ten per cent. nutrient gelatin, preferably made with meat (beef) infusion and Witte's peptone, and brought to a reaction of + 10 (Eyre's scale).

In hot weather it may be necessary to increase the percentage of gelatin.

Some members of the committee advocate the use of meat extracts in place of meat infusion, on the score of convenience and uniformity of composition, Brand's Essence or Bovril's Essence of Meat being recommended as the best. It is the general opinion, however, that *Liebig's Extract* is less suitable for this purpose.

(c) For enumeration at blood heat it is recommended that *nutrient agar* should be employed, being prepared with the same constituents as nutrient gelatin, but substituting $1\frac{1}{2}$ per cent. of powdered agar for the gelatin. Reaction + 10.

(d) *Distilled-Water Agar.*—Powdered agar $1\frac{1}{2}$ per cent., dissolved in distilled water, and brought to a reaction of + 10.

Owing to the changes which occur in the reaction of the medium on keeping, the media employed should preferably be not more than three weeks old.

AMOUNTS TO BE PLATED, SIZE OF DISHES, ETC.

Gelatin.—For an ordinary water amounts of 0·2, 0·3, and 0·5 c.c. may be plated in Petri dishes of not less than 10 centimetres diameter, preferably done in duplicate.

Agar.—Two plates may be made with 0·1 and 1·0 c.c., and are preferably duplicated.

In dealing with an unknown water, and in all cases of doubt, additional sets of plates should be prepared with a dilution of the water (made with sterilized tap-water) of ten or hundred fold, according to circumstances.

The amount of the medium in a plate should be 10 c.c.

The sample must be thoroughly shaken and mixed in all cases before plating.

TEMPERATURE OF INCUBATION.

(a) Room temperature = 18-22° C.
(b) Blood heat = 36-38° C.

COUNTING.

Counting to be done with the naked eye, preferably in daylight, any doubtful colony being determined with the aid of a lens or low-power objective.

Time of Counting.—Gelatin plates should be counted at the end of seventy-two hours; but in all cases the plates should be inspected daily, in order that the count may be made earlier should liquefaction render this necessary.

The blood-heat agar plates should be counted at the end of forty to forty-eight hours.

SEARCH FOR BACILLUS COLI.

Method.—The committee recommend either—

(a) The glucose-formate broth method of Pakes.
(b) The bile-salt broth method of McConkey.

Incubation anaerobically at 42° C. increases the chances of success with either medium, and is strongly recommended.

It has also been suggested that the neutral-red (Grübler's) glucose broth medium may be employed.

The committee do not regard with favour the Parietti method, or the use of carbolic acid media.

QUANTITY OF WATER TO BE EXAMINED.

As a routine 50 c.c. should be the minimal quantity examined for the presence of the *Bacillus coli*, quantities from a minimum of 0·1 c.c. to a maximum of 25 c.c. being added to the tubes of culture media.

The committee are of opinion that it is preferable to add the water directly to the tubes of culture media, even with the larger amounts, rather than first to concentrate by filtration through a porcelain filter (the filter brushing method). The culture media recommended may be diluted with at least an equal volume of the water without interfering with their cultural properties, and large tubes or small flasks may be used for the larger amounts.

In the case of the bile-salt lactose peptone water, the medium may for the larger amounts be prepared of double strength.

ISOLATION OF BACILLUS COLI, IF PRESENT.

If indications of the presence of the *Bacillus coli* be obtained in the preliminary cultivations, the organism must be isolated and identified.

This may be done by making *surface* cultures on plates of either—

(*a*) litmus lactose agar, reaction + 10;
(*b*) bile-salt agar;
(*c*) nutrose agar of Conradi and Drigalski; or
(*d*) ordinary nutrient gelatin.

The best medium of all is, probably, the nutrose agar of Conradi and Drigalski. Agar media have the advantage of saving time.

IDENTIFICATION OF, AND TESTS FOR, THE BACILLUS COLI.

Having obtained coli-like colonies on the plates made from the preliminary cultivations of the water, sub-cultures must be made in order to identify the organism. The following, at least, should be made:

(*a*) Surface agar at 37° C. The abundant growth so obtained enables many sub-cultures and preparations to be made if required.
(*b*) Stab and surface cultures in gelatin. This may be done in the same tube.
(*c*) Litmus milk incubated at 37° C.
(*d*) Glucose litmus medium.
(*e*) Lactose litmus medium.
(*f*) Peptone water for indol reaction.

CHARACTERS OF THE BACILLUS COLI.

The *Bacillus coli* is a small motile, non-sporing bacillus, growing at 37° C. as well as at room temperature. The motility is well observed in a young culture in a fluid glucose medium. It is decolorized by Gram's method of staining. It never liquefies gelatin, and the gelatin cultures should be kept for at least ten days in order to exclude a liquefying bacillus. It forms smooth, thin surface growths and colonies on gelatin, not corrugated, growing well to the bottom of the stab (facultative anaerobe).

It produces permanent acidity in milk, which is curdled within seven days at 37° C. It ferments glucose and lactose, with the production both of acid and of gas.

The typical *Bacillus coli* must conform to the above description and tests.

It generally also forms indol (best obtained in peptone-water cultures), gives a thick yellowish-brown growth on potato (greatly dependent on the character of the potato), sometimes (about 50 per cent.) ferments saccharose, changes neutral red (Grübler's), and reduces nitrates, and half the gas produced by it from glucose is absorbable by KOH; and these tests, if time and opportunity permit, may be performed in addition to the foregoing.

The committee recognise that atypical *Bacilli coli* are met with, but in the present state of our knowledge hesitate to make any suggestion with regard to their significance.

STREPTOCOCCI.

The committee consider that it is a distinct advantage to search for streptococci. They may be looked for by making hanging-drop preparations of the fluid media employed for the preliminary cultivation of the *Bacillus coli* (glucose-formate broth, etc.). The presence or absence of streptococci in these tubes gives also a quantitative value to the examination, just as in the case of *Bacillus coli*, and the result obtained should be stated. The streptococci should be isolated (best carried out on nutrose-agar plates), and their characters determined.

BACILLUS ENTERITIDIS SPOROGENES.

As already stated, the committee do not consider that it is essential as a routine procedure to search for the *Bacillus enteritidis sporogenes*, though in certain instances it may be of advantage to do so. A negative result in such cases is probably of more value than a positive one.

This report is the outcome of prolonged deliberations, and every point has been carefully considered and discussed by the members of the committee.

In conclusion, the committee suggest that if the above recommendations were to be adopted by all engaged in the bacteriological examination of water it would conduce to uniformity of results, and would render comparable the data obtained by different observers. An addendum might be added to a report on an analysis conducted on these lines to the effect that the analysis had been carried out in conformity with the procedures recommended by the committee of The Royal Institute of Public Health, 1904.

The committee beg to acknowledge their great indebtedness to Professor R. Tanner Hewlett, M.D., D.P.H., upon whom the great burden of the work of the committee has devolved.

RUBERT BOYCE, M.B., F.R.S.,
Chairman.

R. TANNER HEWLETT, M.D., M.R.C.P.,
Hon. Secretary.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Treatment of Frying Oils. F. Jean. (*Rev. Gén. Chim. pure et appl.*, 1904, vii., 326; through *Chem. Zeit. Rep.*, 1904, xxiv., 291.)—The author has investigated the cause of frothing of vegetable oils used in cooking, and finds that: (*a*) When the oil is heated to 230° C., the volatile constituents are driven off and the property of "frothing" is lost, while the acidity is reduced. (*b*) When steam-distilled at 150°, the oil froths considerably and an acid distillate is obtained; at 190°-225°, the frothing is reduced and solid particles are carried over; while above 225° the acidity increases. The distillate was found to contain allyl sulphide, aldehydes, and fatty acids. (*c*) When heated with various fruits to 170°, and a current of air passed through the liquid, there was no frothing. H. A. T.

The Determination of Fat in Flesh or Meats by Gerber's Acido-Butyrometer. Toyokichi Kita. (*Arch. Hyg.*, 1904, li., 165; through *Chem. Zeit. Rep.*, 1904, xxiv., 291.)—Gerber's process is perfectly applicable to the determination of fat in meats, etc. After very thorough mincing, the meat is dissolved, and the fat liberated by warming and shaking in the butyrometer with a mixture of equal volumes of water and sulphuric acid (specific gravity 1·820-1·825); amyl alcohol and sulphuric acid are added in the usual way, and the percentage of fat read off after centrifuging for three to five minutes. H. A. T.

The Flash Point or Ignition Temperature of Wine as an Index of the Alcohol Contents. P. N. Raikow and P. Schtarbanow. (*Chem. Zeit.*, 1904, lxxvi., 886.)—In former communications (*Chem. Zeit.*, 1899, xxiii., 145; 1902, xxvi., 437) the authors describe a process for the determination of alcohol in aqueous solution by the flash point or temperature of ignition of the vapour. Abel's apparatus was used, and it was shown that the flash point was governed entirely by the amount of alcohol present; and from a series of experiments reliable tables were drawn up. In applying this method to wines it was found that the presence of certain volatile compounds (as aldehydes, ethers, esters, etc.) sensibly reduced the observed flash point, giving figures indicating larger quantities of alcohol than were actually present. More satisfactory values were obtained by diluting the wine before the determination of the flash point; but as at least 4 per cent. of alcohol is necessary before ignition of the vapour is possible, dilution cannot be carried to any great extent. H. A. T.

The Detection of Saccharin in Wines, etc. Villiers, Magnier de la Source, Rocques, and Fayolle. (*Ann. de Chim. anal.*, 1904, ix., 418-420.)—The liquid is freed from alcohol by distillation and treated with an excess of neutral lead acetate in a slightly acid solution. The excess of lead is removed by precipitation with sulphuric acid, and the acid filtrate extracted with three successive portions of crystallizable benzene, the amount used each time being equal to half the volume of the filtrate. The benzene extract is partially distilled and tested for salicylic acid with a 0·1 per cent. solution of ferric chloride. The distillation is then completed without separating the ferric salt. The aqueous solution in the flask is now treated with 10 c.c. of dilute sulphuric acid (1 : 10) and heated on the water-bath, a warm saturated solution of potassium permanganate being added little by little until the liquid becomes pink. It is next extracted three times with half its volume of benzene, and the combined extracts evaporated on the water-bath. The residue is taken up with 2 c.c. of hot water, and a drop of the solution tasted. If the characteristic intense sweetness be observed, the remainder of the liquid is mixed with 2 c.c. of dilute sodium hydroxide solution (1 part of a solution of 36° with 10 of water) and rapidly evaporated to dryness. The residue is heated for three minutes in a tube immersed in a metal bath kept at 270° C., and then dissolved in dilute sulphuric acid (1 : 10). The solution is shaken with benzene and the extract shaken with 5 c.c. of a 0·1 per cent. solution of ferric chloride. The violet coloration characteristic of salicylic acid confirms the presence of saccharin in the original liquid. C. A. M.

Commercial Substitutes for Pepper. F. Jean. (*Ann. de Chim. anal.*, 1904, ix., 423-425.)—Two powders closely resembling black and white pepper are sold in France under the respective names of *Le Griffon* and *Le Mito.* These are stated to consist of the vetch, popularly known as "*Chère aux pigeons*," and are thus very similar to the pepper substitute sold as *Ervioп* (ANALYST, xxix., 309). An analysis of *Le Griffon* gave the following results: Nitrogenous organic matter, 17·62; fat, 4·16; extractive matters, 62·77; ash, 3·05; and moisture, 12·40 per cent.

C. A. M.

The Constituents of Celery. M. Bamberger and A. Landsiedl. (*Monatsheft. f. Chem.*, 1904, xxv., 1030-1034.)—The root-stalks of fresh plants were found to contain mannite, asparagine, and tyrosine, but the authors could not detect leucine. The amount of asparagine isolated from different samples was fairly constant, 62 grammes yielding about 0·3 gramme, but the proportion of tyrosine was very small. C. A. M.

The Chemical Composition and Formula of Adrenaline. G. Bertrand. (*Bull. Soc. Chim.*, 1904, xxxi., 1188-1193.)—The material used in the author's investigations was extracted from the suprarenal glands of a horse, of which 118 kilos yielded about 125 grammes of crystalline adrenaline. A definite quantity of the alkaloid (110 grammes) was dissolved in 600 c.c. of normal sulphuric acid, and fractionally precipitated by means of ammonia, each of the precipitates being washed with water and alcohol and dried *in vacuo.* The seven fractions thus obtained were found to have the same elementary composition: Carbon, 58·46 - 58·83 per cent.; hydrogen, 7·19 - 7·30 per cent.; and nitrogen, 7·66 - 7·74 per cent. From this the author concludes that adrenaline is a simple substance and not a mixture, and that its formula is $C_{10}H_{13}NO_3$, as found by Aldrich. C. A. M.

The Determination of Antipyrin. P. Lemaire. (*Bull. Soc. Pharm. Bordeaux*, 1904, 225; *Ann. de Chim. anal.*, 1904, ix., 433-435.)—The method is based on the fact that antipyrin combines with picric acid (1 mol. : 1 mol.), forming an almost insoluble crystalline compound. As the precipitate is slightly soluble or dissociates in water, it is necessary to have an excess of picric acid in the solution. The standard solutions required are a $\frac{N}{20}$ solution of sodium hydroxide and a $\frac{N}{20}$ solution of picric acid (11·45 grammes per litre), standardized by titration with the sodium hydroxide solution, with phenol-phthalein as indicator. In the determination 5 c.c. of a 5 per cent. solution of the antipyrin are mixed with 50 c.c. of the standard picric acid solution, which is added little by little with constant shaking. The liquid is then filtered, and 25 c.c. of the filtrate titrated with the standard alkali solution, phenol-phthalein being used as indicator. If n c.c. be required, the proportion of antipyrin is obtained by the formula $(50 - n \times 4{\cdot}4) \times 0{\cdot}0094$, and the result multiplied by 4 gives the amount in 1 gramme of the substance under examination.

The crystalline precipitate can then be crystallized and dried, and its melting-point taken in order to determine whether it contains Lederer's iso-antipyrin, which has the same chemical characteristics and melting-point as Knorr's antipyrin, but is poisonous. The difference in the melting-points of the respective picrates affords a means of distinguishing between the two, the picrate of iso-antipyrin melting at 168° C., whilst that of true antipyrin melts at 187° C.

A colorimetric method can also be used in the determination, the filtrate from the insoluble compound being compared with solutions of picric acid of known strength.

C. A. M.

ORGANIC ANALYSIS.

The Estimation of Benzene Vapour in Coal Gas. Otto Pfeiffer. (*Chem. Zeit.*, 1904, lxxvi., 884.)—Harbeck and Lunge's method for the determination of benzene in coal gas, which depends on the passage of the gas through a mixture of nitric and sulphuric acids with consequent nitration of the benzene and separation of this as di-nitrobenzene, is unnecessarily complicated. The gas is only measured after passing the acids, which necessitates special determinations of CO_2 and of the heavy hydro-carbons.

In the author's modification the whole reaction is conducted in one piece of apparatus, and the di-nitrobenzene is titrated with stannous chloride.

A 500 c.c. separating funnel, of accurately known capacity, is filled with the gas to be examined. The funnel is inverted, and 2 c.c. of mixed sulphuric and nitric acids (equal volumes of concentrated H_2SO_4 and fuming HNO_3) are introduced into the outlet tube, and carefully run into the separating funnel. The acids are distributed as much as possible over the inner surface, and the funnel allowed to stand. After half an hour 30 c.c. of concentrated soda solution are introduced in the usual manner, and the funnel shaken until the vapour formed disappears. If still acid the solution is neutralized with soda, and again rendered just acid with HCl, the nitro-compound extracted by repeated shaking with small quantities of ether, and the ethereal solution brought into a small flask with 1 gramme of dry potash and about ½ gramme of animal (blood) charcoal. After shaking the solution is filtered into a 200 c.c. flask, washed with ether, and this entirely driven off on the water-bath. About 10 c.c. of alcohol and exactly 10 c.c. of stannous chloride solution (150 grammes tin dissolved in hydrochloric acid + 50 c.c. HCl made up to 1,000 c.c. with water) are added, and the flask heated on the water bath for ten minutes. The solution is then made up to the 200 c.c. mark, and 20 c.c. are titrated with $\frac{N}{10}$ iodine, using starch as indicator.

The reaction between the stannous chloride and the di-nitrobenzene takes place according to the equation :

$$C_6H_4(NO_2)_2 + 6SnCl_2 + 12HCl = C_6H_4(NH_2)_2 + 6SnCl_4 + 4H_2O.$$

A blank experiment is made by heating 10 c.c. of stannous chloride solution with alcohol, diluting to 200 c c., and titrating 20 c.c. of this as above. H. A. T.

The Composition of Borneo Tallow. J. Klimont. (*Monatsheft. f. Chem.*, 1904, xxv., 929-932.)—This fat is obtained from the fruit of different varieties of *dipterocarpus*. The specimen examined by the author was a hard, yellowish-green fat, and gave the following analytical results: Acid value, 15·8; saponification value, 194·6; iodine value, 30·1; and melting-point, 34·5° to 35·7° C. It was found to contain tristearin with (probably) tripalmitin and at least two mixed glycerides. By repeated recrystallization of the fat (after removal of free fatty acid) from acetone crystals with the double melting-points 44° C. and 37° C. were obtained, and the elementary analysis and saponification and iodine values of these showed them to consist of oleo-distearin, $C_3H_5.(C_{18}H_{33}O_2).(C_{18}H_{35}O_2)_2$. Another fraction yielded oleo-dipalmitin, $C_3H_5.(C_{18}H_{33}O_2)(C_{16}H_{31}O_2)_2$, melting at 33° to 34° C., and at 28° to 29° C. after being once melted. C. A. M.

Investigation of the Colour Reactions of Wood Fibre. V. Grafe. (*Monatsheft. f. Chem.*, 1904, xxv., 987-1029.)—The yellow coloration given by woody fibre with aniline salts, the red coloration with phloroglucinol and hydrochloric acid, and colorations given with other phenols are grouped by the author under the term of "Wiesner's reactions," and attributed to the action of certain constituents in the wood fibre upon these respective substances, while the hydrochloric acid only plays the part of accelerating agent. From his experiments (described in detail) he concludes that wood fibre contains several constituents, the principal being vanillin, methyl-furfural, and pyrocatechol, together with coniferin partly in the free state and partly combined with the cellulose as an ester and in the resin. The blue coloration given by wood fibre in the test with phenol, hydrochloric acid, and potassium chlorate is to be attributed to the coniferin, while the green coloration with concentrated hydrochloric or hydrobromic acid is most probably due to the methyl-furfural in conjunction with the coniferin. In the author's opinion, Wiesner's reactions cannot be regarded as certain tests of lignification, since it is possible to prepare pyrocatechol and methyl-furfural in a simple way from cellulose, whilst the vanillin in the ligneous membrane may have had a similar origin. Maule's reaction, which consists in treating chips of wood for five minutes with potassium permanganate solution (1 per cent.), washing them with water, immersing them in hydrochloric acid to remove the manganese dioxide, and finally exposing them to ammonia fumes (reddish-brown coloration), is also to be attributed to the presence of the same constituents that produce Wiesner's reactions. C. A. M.

A New Reagent for the Detection of Blood Colouring Matters and their Decomposition Products. E. Riegler. (*Zeit. anal. Chem.*, 1904, xlii., 539-544.)—The method is based upon the formation of an alkaline alcoholic solution of hæmochromogen, which has a brilliant purple red colour, and shows with great clearness the two characteristic absorption bands in the spectrum. The reagent is prepared by shaking a solution of 10 grammes of sodium hydroxide in 100 c.c. of water with 5 grammes of hydrazine sulphate until the latter dissolves, and then adding 100 c.c. of 96 to 97 per cent. alcohol. The liquid is well shaken, allowed to stand for two hours, and filtered. The filtrate gives the same hæmochromogen reaction with blood, oxyhæmoglobin, hæmoglobin, methæmoglobin, or hæmatin. Thus, on shaking 0·05 gramme of commercial hæmoglobin or 0·5 c.c. of blood with 30 c.c. of the reagent, the characteristic coloration is obtained. In testing liquids such as urine for blood 10 c.c. are shaken with 10 c.c. of the reagent, and the whole allowed to stand for thirty minutes. For the detection of blood stains on linen, a small fragment of the material is moistened with 3 or 4 drops of the reagent, and examined under the microscope, and eventually with the microspectroscope. C. A. M.

INORGANIC ANALYSIS.

Electrolytic Determination of Bismuth. A. Hollard and L. Bertiaux. (*Bull. Soc. Chim.*, 1904, xxxi., 1131-1133.)—*Separation of Bismuth and Copper.*—The solution of the two metals in the form of sulphates (which should not contain a large excess of sulphuric acid) is brought to the boiling-point, and treated with a large excess of phosphoric acid. After standing overnight, the precipitate is collected and washed with dilute phosphoric acid (1 part, of specific gravity 1·711, with 19 parts of water), and then with ammonium hydrosulphide and potassium cyanide solution to remove the copper. This washing solution ought to contain in 100 c.c. 5 grammes of potassium cyanide and 5 c.c. of ammonium hydrosulphide, obtained by saturating a 10 per cent. solution of ammonia with hydrogen sulphide. The precipitate of bismuth phosphate is dissolved in dilute nitric acid (1 : 1), and the solution evaporated with 12 c.c. of sulphuric acid until abundant white fumes appear, when the bismuth will have been converted into pyrophosphate. The residue is then diluted to 300 c.c., and the liquid electrolysed for twenty-four hours with a current of 0·1 ampère per square decimetre. (No other details given.) The bismuth deposited should be tested with ammonia for copper, which, if found, should be determined colorimetrically.

Separation of Bismuth and Lead.—The separation of lead from bismuth in the form of sulphates is not accurate when the proportion of lead is large, owing to the lead sulphate retaining a considerable amount of bismuth; whilst if a solution of bismuth in sulphuric acid containing insoluble lead sulphate be electrolysed, an appreciable quantity of lead is deposited with the bismuth. This difficulty is obviated as follows: The solution containing the two metals in the form of nitrates is evaporated with an excess of 12 c.c. more than the amount of sulphuric acid required to combine with the bismuth and lead present. As soon as abundant white fumes appear the liquid is cooled, diluted to 300 c.c., mixed with 35 c.c. of absolute alcohol, and electrolysed for forty-eight hours with a current of 0·1 ampère. The addition of the alcohol renders the lead sulphate completely insoluble without interfering with the deposition of the bismuth. The results quoted show that minute quantities of bismuth can be accurately separated from large amounts of lead by this method.

C. A. M.

Analysis of Commercial Tin and its Alloys. A. Hollard and L. Bertiaux.

(*Bull. Soc. Chim.*, 1904, xxxi., 1128-1131.)—*Arsenic.*—Five grammes of the tin are placed in an apparatus for distillation of the arsenic, and treated with a solution of 50 grammes of ferric sulphate in 150 c.c. of hydrochloric acid, and the arsenious chloride determined volumetrically (Analyst, xxv., 301).

Lead, Bismuth, and Iron.—Five grammes of the tin are dissolved in hydrochloric acid, with the addition of the smallest possible quantity of nitric acid. Ammonia is then added in excess, after which a current of hydrogen sulphide is introduced, until the whole of the tin has dissolved. The solution is filtered from the sulphides of lead, iron, and bismuth, and the part of the copper sulphide that has not been dissolved by the ammonium sulphide. The precipitate is washed with water, and then with a solution of hydrogen sulphide, and redissolved in hydrochloric acid containing bromine. The excess of bromine is expelled by heat, and the liquid treated with hydrogen sulphide, which precipitates the lead, bismuth, copper, and the small amount of tin which was precipitated with them. This precipitate is collected and washed with a solution of hydrogen sulphide.

The iron left in the solution is precipitated with ammonia after the liquid has been boiled and treated with nitric acid. The precipitate is redissolved in hydrochloric acid, reprecipitated with ammonia, and the iron determined volumetrically by converting it into ferric chloride, nearly neutralizing the solution with sodium bicarbonate, mixing it with 5 c.c. of carbon bisulphide and an excess of a concentrated solution of potassium iodide, and leaving it in the closed flask for thirty minutes, after which the liberated iodine is titrated with standard sodium thiosulphate solution—

$$Fe_2Cl_6 + 2KI = 2FeCl_2 + 2KCl + 2I.$$

The precipitate of sulphides is dissolved in hot hydrochloric acid containing bromine, solution being completed by washing with hot water. The liquid is mixed with ammonia in excess and a little potassium cyanide (to keep the copper in solution), and treated with a current of hydrogen sulphide, which now precipitates only the lead and bismuth. The precipitate is collected, washed with dilute ammonium hydrosulphide, then with water, and then with a solution of hydrogen sulphide. The sulphides are converted into sulphates by being dissolved in nitric acid containing bromine and evaporation with sulphuric acid. The lead sulphate is filtered off and determined electrolytically (*Bull. Soc. Chim.*, xxxi., 239), whilst the bismuth is determined electrolytically in the filtrate (*Comptes Rendus*, 1904, 366).

Antimony.—One gramme of the original tin is treated with *aqua regia*, and the nitric acid subsequently expelled by repeated evaporation with hydrochloric acid. The dry residue is rendered alkaline by the addition of several drops of sodium hydroxide solution, and taken up with a mixture of 200 c.c. of concentrated sodium hydrosulphide solution and 40 c.c. of a 20 per cent. solution of potassium cyanide, and electrolysed with a current of 0·1 ampère, only the antimony being deposited (Analyst, xxviii., 228).

Copper and Sulphur.—Five grammes of the tin are treated with nitric acid, and the mixture evaporated to dryness on the water-bath. The residue is taken up with water acidified with nitric acid, the liquid decanted, and the residue washed with the same acidified water. The copper in the filtrate and washings is separated and determined by electrolysis. The sulphur in the solution is determined by evaporating the liquid on the water-bath, taking up the dry residue with water, filtering the solution, adding a few drops of hydrochloric acid, and precipitating the sulphate with barium chloride.

Composition of Commercial Samples.—The following results were thus obtained :

Commercial Tin.	Copper.	Arsenic.	Antimony.	Nickel and Cobalt.	Iron.	Zinc.	Lead.	Bismuth.
	%	%	%	%	%	%	%	%
German	0·480	0·079	0·545	—	—	—	—	0·060
Swiss	—	0·022	1·174	—	—	—	0·498	0·580
Commercial sample ...	0·000	0·034	0·110	0·028	0·040	—	0·076	—
Chinese	0·040	0·118	0·044	—	0·037	—	0·833	—
Malaccan	0·030	0·033	—	0·014	0·008	—	0·081	—

Alloys of Tin and Lead (Solders, etc.).—One gramme of the finely-divided alloy is mixed with 10 grammes of copper, and treated with 52 c.c. of nitric acid in a 350 c.c. beaker. The nitric acid used is diluted with a little water, the amount of which is smaller in proportion to the quantity of tin present. The liquid in the beaker is diluted to 300 c.c., and heated on the water-bath for a short time to make the oxide of tin collect at the bottom, after which it is cooled and the lead separated electrolytically in the form of peroxide (*Bull. Soc. Chim.*, 1904, 293). The spiral immersed in the liquid ought to reach nearly to the bottom of the flask. Under these conditions the oxide of tin does not interfere with the separation of the lead.

C. A. M.

An Improvement on Drown and Shimer's Methods of determining Silicon in Iron. J. Thill. (*Zeit. anal. Chem.*, 1904, xliii., 552, 553.)—An objection to these methods is the long time required for the evaporation of acids used, whilst if a naked flame be used instead of the water-bath for heating, there is danger of loss. The following modification is stated to give accurate results within an hour. From 1 to 2 grammes of the powdered crude iron (according to the amount of silicon) are placed in a beaker holding 400 to 500 c.c., and treated with 50 to 70 c.c. of a solution prepared as follows : A litre of concentrated sulphuric acid is diluted with an equal volume of water, and mixed with a litre of nitric acid (specific gravity 1·40) and a solution of 240 grammes of ammonium chloride in a litre of water. After adding the required amount of this reagent to the iron, the liquid is heated to dissolve the iron, and evaporated on wire gauze over a Bunsen flame until white fumes appear. The residue is then diluted with about 100 c.c. of water, and heated until the sulphates have completely dissolved, after which the liquid is filtered, and the insoluble residue washed with hot water, then with 10. c.c. of hot dilute hydrochloric acid, and again with hot water. Finally, the filter and its contents are ignited while still moist, then heated in a muffle, and weighed. C. A. M.

The Colorimetric Determination of Carbon in Steel. H. C. Boynton and H. K. (*Stahl und Eisen*, 1904, 1070; through *Chem. Zeit. Rep.*, 1904, xxiii., 283.)—The condition of the carbon in steel determines the amount of colour produced in Eggertz process.

Three samples were taken of a piece of steel containing 0·48 per cent. of carbon, and heated to 1200° C. The first was cooled rapidly in an air blast, the second was allowed to cool spontaneously in the air, while the third was left in the furnace and cooled very gradually. The analysis of the blast-cooled sample showed only 0·34-0·35 per cent. of carbon, that of the air-cooled sample 0·36-0·39 per cent., while the sample cooled in the furnace gave the full (0·48 per cent.) amount. H. A. T.

APPARATUS.

Note on the Durability of Platinum Crucibles. G. Siebert. (*Chem. Zeit.*, 1904, lxxiv., 869.)—The author, who is a member of Messrs. G. Siebert's Platinum Works, Hanau, considers that the occasional rapid deterioration and fracture of platinum crucibles, etc., is not always due to the presence of chemical impurities, such as iridium, but rather to the physical properties of the platinum. He finds that when proper precautions are taken to obtain the highest possible degree of homogeneity in the platinum in all stages of manufacture, the crucibles are practically indestructible. Where, however, such precautions are not taken, though they may be made with pure metal, deterioration is very rapid in the articles. H. A. T.

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OBITUARY NOTICE.

THOMAS A. POOLEY, B.Sc., F.I.C.

We regret to record the death, after a prolonged illness, of Mr. Thomas A. Pooley, B.Sc., F.I.C., for many years Public Analyst for the County of Essex and the Borough of West Ham, and an old member of the Society. Mr. Pooley, who was sixty-three years of age at the time of his death, was educated first in France and subsequently at University College and King's College, London. He took an honours science degree at London University in 1864, with special distinction in chemistry. For a number of years he directed his attention chiefly to the chemistry of brewing, being for a time Editor of the *Brewers' Guardian* and for a number of years a regular contributor to the *Brewers' Journal*. During the later part of his life he devoted himself chiefly to food analysis in connection with the duties of his public appointments already referred to. He rarely attended the meetings of the Society, and was therefore personally unknown to very many of his colleagues, but by the few who knew him his kind and genial friendship was much prized.

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FEBRUARY, 1905. Vol. XXX., No. 347.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A SPECIAL meeting of the Society was held on Wednesday evening, January 11, in the Chemical Society's Rooms, Burlington House. The President, Mr. Thomas Fairley, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. W. Ackroyd and J. H. Johnston were read for the second time.

Messrs. P. Edgerton, J. McLeod, G. A. Pingstone, and A. W. Thorp were elected members of the Society.

A discussion, opened by Mr. Hehner, took place on the subject of "Brandy."

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ELECTRIC FURNACES FOR LABORATORY USE.

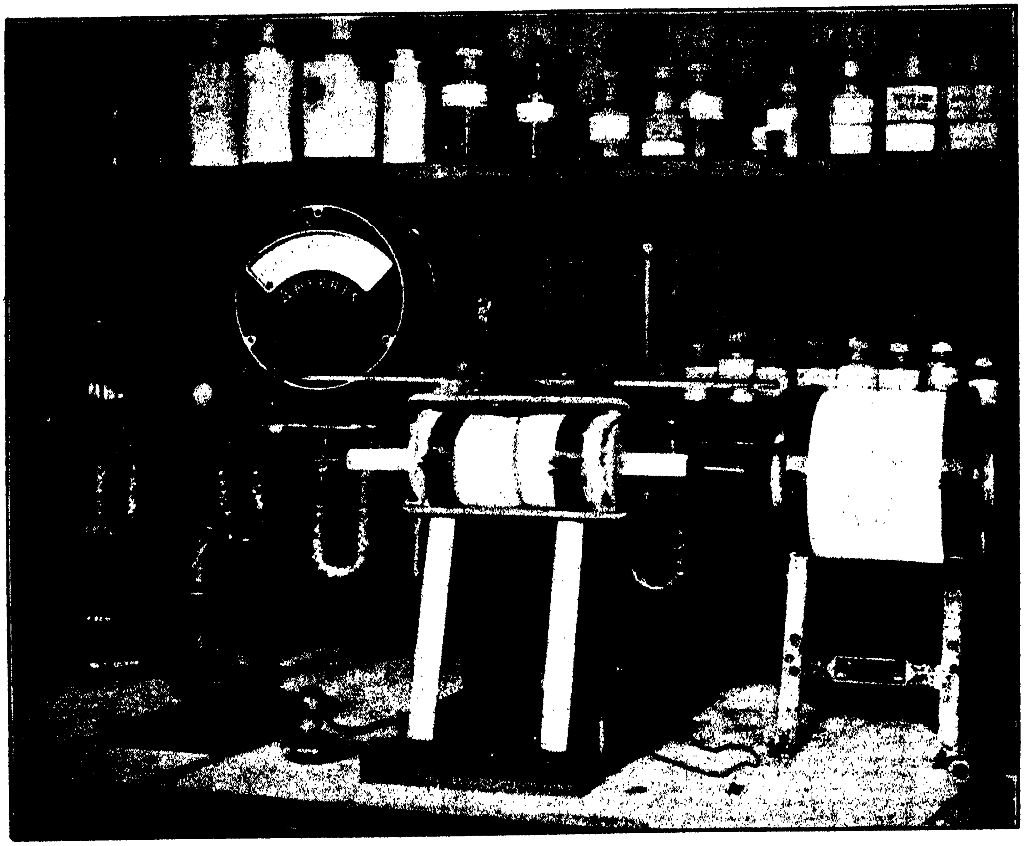
By BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, December* 7, 1904.)

HEATING by gas is not always convenient for laboratory use, because it is difficult to apply the heat so exactly that the object to be heated receives the greater part of the heat. If the thermal units generated by an ordinary Bunsen burner be computed, and those strictly necessary to raise the temperature of the object to be heated be reckoned, it will be found that the efficiency of the arrangement will be of the order of magnitude of 1 per cent. This would be a small matter, were it not that the whole of the gas thus wasted produces its proper quantities of water, carbonic acid and the like, and fouls the atmosphere of the laboratory to a corresponding extent. When a high temperature is needed the gas must be consumed at a greater rate, and a blast is necessary. This requires bellows or some form of blower, and these are troublesome and noisy.

The peculiar quality of electric heating is the ease with which it may be directed to the place where it is needed and the precision of its regulation. The object to be heated may be raised to any determined temperature, and this temperature may be maintained indefinitely. Seeing that a sufficient current put through a suitable resistance will heat the resistance and anything with which it may be in contact to a temperature dependent solely on the current and the drop of voltage between the points where the current enters and leaves the resistance, it would appear to be a simple matter to devise a furnace for laboratory use.

But, as generally happens in practical affairs, little difficulties crop up. The obvious method of heating a tube, crucible, or muffle by wrapping round it fine platinum wire and passing an appropriate current, fails, for the reason that unless the temperature is very moderate the wire will be heated to a point where it is ductile, and the necessary slight tension on it will draw it down in places. These attenuated points will become hotter than the rest of the wire, and will finally fuse. The difficulty of using wire is much increased when, as is usual, the current to be employed is supplied at a moderately high pressure—*e.g.*, 200 volts. In this case the section of the wire must be small, and the risk attending its reduction in section, consequent on some casual tension, will be proportionally greater. This difficulty has been met and overcome by Heræus of Hanau, who conceived the excellent idea of substituting for the wire a strip of platinum foil. The section of this is, naturally, the same as that of a wire to convey the requisite current, but the fact that it is flat allows it to sit close upon the surface to be heated. Hence it will not stretch and



break as will a wire, because it is well supported by the surface on which it is wrapped and also because the close contact of all its parts with that surface allows the transference of heat to take place so rapidly that the temperature of the heating-strip is never much higher than that of the object to be heated. These simple considerations have been philosophically applied by Heræus. His apparatus consists of a tube of specially refractory porcelain, the so-called Macquart's Masse made by the Royal Porcelain Works of Charlottenburg, round which is wrapped a strip of platinum foil so thin that it may be broad, and yet possess the requisite resistance. The strip is 13 millimetres in width for 100 volts and 7 millimetres for 200 volts and 0·007 millimetre in thickness. A tube furnace 20 centimetres in length requires only a small weight of platinum; for example, 6 grammes.

In Heræus's apparatus the resistance is wrapped round a tube, which serves as a muffle for an internal tube. It can be used for all ordinary tube-furnace purposes, such as the ultimate analysis of organic substances and the combustion of carbon in steel. The temperature can be raised to any point short of the fusion of the platinum and the porcelain, and its regulation is easy. The apposition of the platinum to the porcelain is so good that fracture of the former is not likely to occur. In short, this apparatus could scarcely be bettered if it were not somewhat slow in cooling; in consequence, successive operations occupy a relatively long time. This slowness is due chiefly to the excellent non-conducting sheath of the furnace. If the envelope were less efficient, some waste of current would occur, but would be more than balanced by greater speed of working.

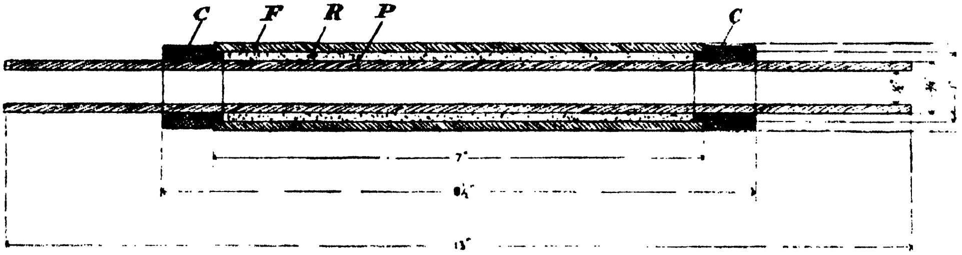


FIG. 1.—SECTION OF "DIRECTLY-PACKED" FURNACE.
C, *C*, Copper end plates; *F*, fireclay or porcelain outer tube; *R*, resistance mixture; *P*, porcelain inner tube.

It seemed possible to construct an effective resistance furnace on somewhat different lines from those adopted by Heræus. In my own work I required a furnace for the determination of oxygen in copper, which could be rapidly heated to the fusing-point of the metal. Mr. Owen Lucas was good enough to suggest that it might prove practicable to prepare an annular resistance of retort carbon which would serve to heat a porcelain tube. The idea was put into working shape in my laboratory, and finally took the following form:

The first form of furnace was one in which the porcelain tube to be heated was surrounded with a similar but rather wider tube, and the annular space between them was packed with a resistance material.

A porcelain tube (Fig. 1) ¾ inch external diameter was placed inside a shorter and wider porcelain or fireclay tube, about 1 inch in internal and 1¼ inches in external diameter, and 7 inches long, so that about 3 inches of the inner tube projected at each end. The space between the two tubes was filled with the resistance material, consisting of retort carbon mixed with varying percentages of siloxicon or carborundum, the current being conducted to this material by two short tubes of copper which fitted closely into the space between the two tubes. This type of furnace required a maximum current of 6 ampères.

Afterwards it was found that the muffle principle used by Heræus was preferable, and a furnace of this type was constructed. (Fig. 2.)

The essential parts of this latter form of furnace are two concentric fireclay tubes, surrounding, but not actually touching that part of the porcelain tube to be heated. The resistance material is packed between these two fireclay tubes, the current being led in by means of an annular copper disc at each end, which is provided with a short cylindrical projection, fitting closely in between the two tubes, so as to make good contact with the resistance material. If the space between the two tubes is very narrow, this may consist of pure retort carbon only; otherwise, for wider spaces, which take more of the material, its resistance may be increased by adding varying amounts of siloxicon. Carborundum has also been used for this purpose, but does not answer as well as siloxicon, as its resistance falls extremely rapidly at high temperatures, apparently approaching that of the carbon. The two tubes which form the furnace proper are surrounded by two other wider fireclay tubes, which serve merely to insulate the heat. They may be covered with asbestos wrapping still further to retain the heat. The ends of the furnace are made of uralite discs, which also support the porcelain tube to be heated.

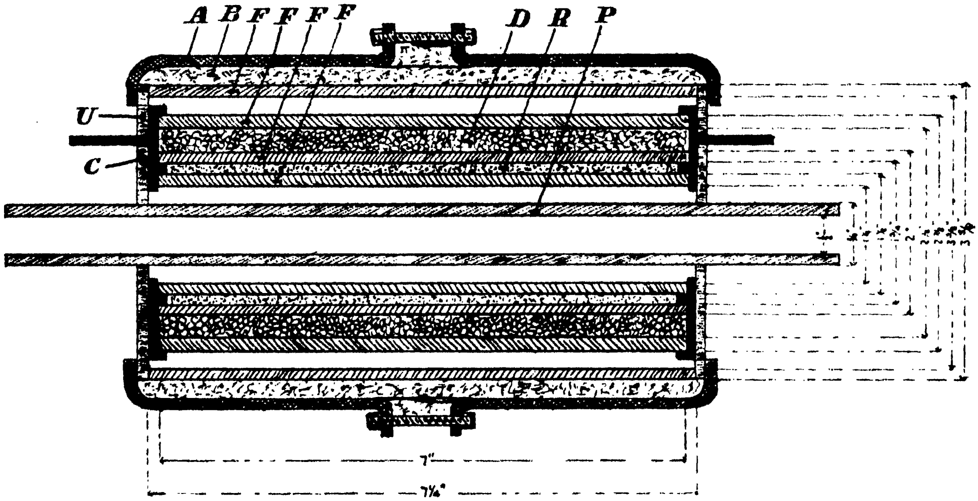


FIG. 2.—SECTION OF "INDIRECTLY-PACKED" TUBE.

A, Clamps ; *B*, asbestos cloth ; *C*, copper terminals ; *F*, fireclay tubes ; *D*, pieces of fireclay ; *R*, resistance mixture ; *P*, porcelain tube ; *U*, uralite discs.

With this apparatus a piece of copper weighing 15 to 20 grammes can be fused in fifteen minutes; and after its fusion and the reduction of the oxide it contains the whole contrivance is cool enough to allow of the removal of the copper and the insertion of another sample in the space of twenty minutes. With a gas furnace the time occupied is much the same, but the surplus heat and waste gases are serious drawbacks.

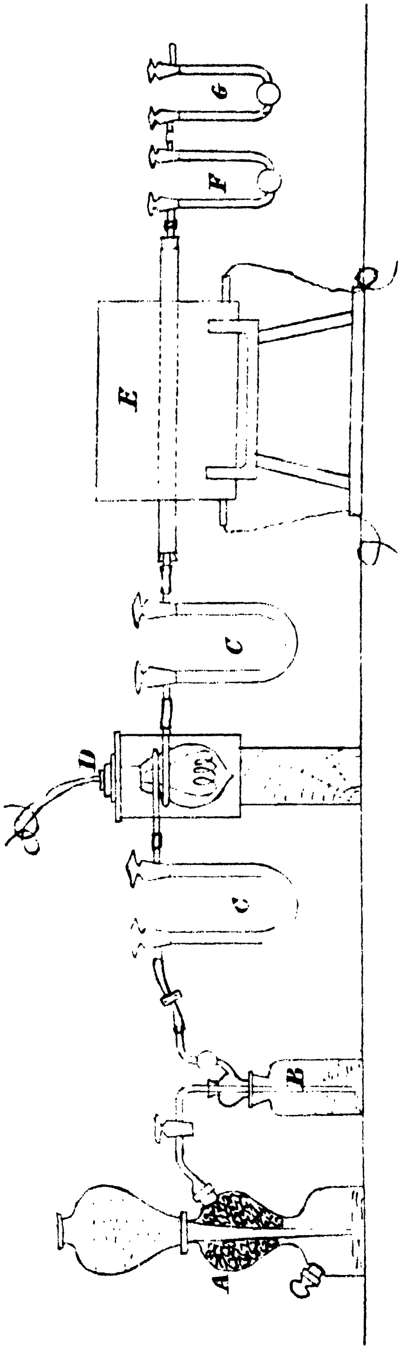


FIG. 3.—APPARATUS FOR DETERMINING OXYGEN IN COPPER.

A, Hydrogen generator; *B*, wash-bottle; *C*, *C*, sulphuric acid drying tubes; *D*, copper oven for heating spiral containing tinised asbestos by means of a glow lamp; *E*, furnace; *F*, weighed sulphuric acid tube; *G*, guard tube.

In the description given some time ago (ANALYST, xxi., 57) of my method of determining oxygen in copper the details of working are described; one of these is the purification of the hydrogen destined to reduce oxides in the copper by passing it over heated platinized asbestos. For convenience' sake, and to make the apparatus independent of clumsier methods of heating, this, too, has been done electrically. The platinized asbestos is contained in a tube, which is coiled round a glow lamp and protected by an insulating sheath. It is kept hot enough to induce the combination of any small quantity of oxygen that may be present in the hydrogen. Formerly, when gas was used, this essential precaution had to be secured by a bulb-tube, heated by a Bunsen burner. The difference in point of convenience is considerable. The apparatus as set up for use is shown in Fig. 3, and the photograph on p. 30.

I have described a particular use of a particular form of electric furnace for laboratory purposes. The use may be extended to many operations, and the form may be modified accordingly. The fundamental idea is the adoption of a resistance material, which can be packed into an annular space between two porcelain tubes, and the resistance of which can be regulated by its cross-section and by its composition, which is so ready to hand and so cheap that any required furnace can be prepared at short notice.

It seems to me that almost all common laboratory heating operations would be best done electrically; the chemist himself can hardly be expected to devise detailed methods, but the instrument-maker might well try.

I desire to thank Mr. A. G. Levy and Mr. H. E. Course for their valuable aid.

DISCUSSION.

The PRESIDENT (Mr. Fairley) said that a simple method of electrically heating a liquid—namely, by the immersion in it of an incandescent lamp—had been recently described before the Nottingham section of the Society of Chemical Industry (*Journal of the Society of Chemical Industry*, 1904, xxiii., p. 1137). The method was particularly applicable to distillation. Electricity would, no doubt, in the future be widely used for heating purposes. In his own district only an alternating current was supplied. This, however, could be used with special appliances, or could be transformed.

Mr. RICHMOND inquired whether Mr. Blount had used an alternating current in any of his experiments. The heating effect of the alternating current would be the same as that of the direct current, but the direct current arc was far more destructive than the alternating current arc, and one would require to pay more attention to the contacts.

Mr. L. MYDDELTON NASH asked whether the tube containing the boat could not itself be surrounded by the platinum foil, thus avoiding the use of two tubes, and consequent loss of available heat.

Mr. CHAPMAN asked whether, with the furnace shown, it was easy to arrange for progressive, distributed, or divided heating, such as was of course necessary in ordinary ultimate organic analysis. For some years he had been making use of electric water-baths in his laboratory for the distillation of ether and other inflammable liquids with the greatest possible advantage.

Mr. SUTHERLAND said that he would have liked to hear more about Mr. Blount's experience with siloxicon with regard to its general properties, and use for lining large furnaces, cost, etc. It was said to be harder than carborundum, than which it was, he believed, somewhat dearer. Its price was about 4d. per pound. He would also like to ask Mr. Blount whether the principles of the furnaces he had described had been successfully applied to the heating of furnaces for crucibles and muffles or larger furnaces. There should be a large field in this direction for electric heating.

Mr. BLOUNT said that he must confess some ignorance with regard to the use of alternating currents for heating. He believed that alternating currents had been tried in these furnaces, and that the difference in their operation with direct and with alternating currents was small. The number of watts being the same, the heat generated must be the same, and one would hardly expect those differences which might be observed in the case of arcs to be reproduced in heating through a resistance. The only possible alteration in working that could be foreseen would be some difference at the points of contact of the terminals with the resistant material, or of the resistant material itself, and clearly that difference, if it existed, would apply only to his form of furnace, and not to that of Heræus, in which the conductor was continuous. With regard to the use of a single tube, perhaps in going through the paper he had not been sufficiently explicit. He had meant to make it clear that the furnace, as he had modified it, was originally a single-tube furnace, the heating being direct on the tube it was desired to heat. When a platinum conductor was used, a single tube was not efficient, because for ordinary operations with gases it must be glazed, and the platinum foil, if wound on a glazed tube, ate into the glaze and was soon destroyed. The tube must be biscuit porcelain, which was not impervious, so that the porcelain tube must be used as a muffle. In the second form of his (the author's) furnace a similar muffle was adopted, using fireclay instead of biscuit porcelain. The furnace was quite capable of being broken up into divisions to secure the distributed effects mentioned by Mr. Chapman. Indeed, Heræus had already made furnaces in sections, and in such furnaces ultimate analyses could be performed as easily as in an ordinary furnace, though the process was somewhat slower.

Mr. SUTHERLAND asked whether a tube glazed on the inside only could not be used.

Mr. BLOUNT said that he had tried the use of a tube glazed on one side, but had not found it to be satisfactory. Mr. Sutherland had asked about siloxicon, which was a very interesting substance. It was not conspicuous for its hardness: its real value lay in its refractoriness. As a furnace lining it was considerably used, and, although not so stable at high temperatures as carborundum, was well adapted for lining furnaces, which were subjected not only to high temperatures but to the action of reducing or oxidizing agents. It was being employed as a protective lining for rotatory cement kilns, which were subjected to as heavy a wear as any sort of furnace. As to its price, he could not say much, but it must be less than that of carborundum, which was very moderate—about £20 a ton. The price of 4d. per pound mentioned by Mr. Sutherland would therefore seem to have been a retail one. He agreed with Mr. Sutherland in thinking that there was a large field open in the direction of large furnaces and furnaces for crucibles. He thought, however, that the devising of such appliances might safely be left to the instrument makers. The road into that field had been opened by Heræus, and, in a smaller degree, by his own work.

In reply to further questions put by Mr. HEHNER and by Mr. SUTHERLAND, Mr. BLOUNT said that in the analysis he had made of the siloxicon, the carbon had been determined by combustion with lead chromate. The silicon had been directly determined, and the oxygen was by difference. The siloxicon, however, was not likely to burn away when used as a furnace lining: nothing short of actual fusion with a substance like lead chromate would affect it.

* * * * *

BRANDY.

By OTTO HEHNER.

(*Read at the Meeting, January* 11, 1905.)

On November 29, 1902, there appeared, as a supplement to the *Lancet*, a report of a Special Analytical Commission on "Brandy: its Production as Cognac, the Present Position of the Industry in the Charentes, and the Supply of Genuine Brandy to this Country." This report appears to have been the stimulus which caused certain Public Analysts to take up the examination of commercial brandies under the Sale of Food Acts. Although the report of the *Lancet* Commission contained not only nothing that should have been novel to any well-read Public Analyst, but gave much less information than had been obtainable for at least ten years previously, it seems to have directed attention to what is undoubtedly a most important question. It is our business as Public Analysts to carry out the provisions of the Sale of Food and Drugs Acts, and to see that when the public demands any article of food or drink, an article of the "nature, substance, and quality demanded" is supplied. It is equally our business as scientific men to act up to the scientific knowledge available, but not to go one step beyond, and pretend to do that which is in our present state of knowledge impossible.

It will be seen that the *Lancet* report, although dealing with brandy, concerned itself solely with its production at Cognac and with the position of the brandy industry in the Charentes, the home of the Cognac industry. That is to say, it dealt with that variety of brandy which is known as "cognac." If all brandy were cognac, if only that which is produced in the Cognac district and by the Cognac methods had the right to be called "brandy," the matter would be a comparatively simple one. We are all agreed that the term "cognac" applies only to a distillate obtained from wine, and from nothing else, containing the full flavour peculiar to cognac—a flavour only obtainable in a product resulting in a more or less primitive manner by pot-still distillation, without much fractionation or purification. Opinions are, and may be legitimately, divided on the question whether "cognac" nowadays belongs only to the product obtained, as described, in the Cognac district itself, or whether the term has lost its geographical significance, as "Munich beer," "Yorkshire pudding," or "Bath bun" have.

The term "brandy" is obviously not synonymous with "cognac," for in the immense wine industry—one of the greatest agricultural industries of the world—wine distillates are obtained which could not possibly be classed or sold as "cognacs," and yet possess wine flavour or bouquet. Such distillates are known in this country as "brandy." I take this term to be a corruption of the German word "Branntwein," literally "burnt wine"—a wine distillate. Many generations ago, before Continental nations had learned to prepare distilled alcoholic beverages from grain or other starchy material, all "Branntwein" was a wine distillate, whilst now it only means a distilled alcoholic beverage mostly made from grain, but not necessarily from that only. It corresponds to "eau de vie" of the French, or the "fire-water" of the Red Indian. But while both the French and Germans were satisfied with the gradual drifting of the respective terms "eau de vie" and "Branntwein" to beverages not necessarily made from wine—just as we apply the word "vinegar" no longer to acidified wine, but to acid liquors of other derivation—the English purchaser, as of old, attached the term "brandy" to the wine distillate only. Brandy not being an English production, it is perhaps natural that he should remain behind in the knowledge that the industry was changing.

I will not for the moment stop to consider whether the English purchaser is reasonable in attaching a meaning to a term which it no longer possesses in other countries, but will for the present put myself upon the standpoint of the purchaser, only expressing my opinion that his view is old-fashioned, narrow-minded, and unreasonable. In the case of the national distilled alcoholic beverage "whisky"—which once upon a time was the liquor obtained from fermented malt-wort only, distilled from pot-stills—the nation has not objected to the widening of the term, substances other than malt being used, and scientific rectification stills having largely taken the place of the old pot-stills. Differences in flavour and in composition have, of course, followed the alteration in manufacture. In the case of beer the changes are wider still; there is not, in fact, a manufactured article of food or drink where changes in preparation and in composition are not continually being made. Brandy, for the purposes of my argument, shall form an exception, for the purchaser, however ignorant and prejudiced, is entitled to his prejudice, and the vendor must supply him with "the article demanded."

We analysts have no other means than chemical, or physico-chemical, of ascertaining whether an article is "genuine" or not, and for us the whole question is an analytical one. We obviously cannot distinguish pure alcohol derived from wine from that derived from the potato, any more than we can differentiate between sugar made from the cane and that made from beet. We have nothing to go by but the concomitants of the alcohol, the "impurities" which give to the alcohol its aroma, flavour, and character. Apart from solid substances that have been taken up from the wood of the casks, or that have been intentionally added for colouring or sweetening, the "impurities" of spirits belong to five or six *classes* of bodies—alcohols other than ethylic; acids, mainly acetic; aldehydes, chiefly ethylic; ethers; basic substances; and furfural. Every one of these classes contributes its quota towards the character of the beverage, probably also towards its physiological properties.

Since 1883, when Nessler and Barth correctly stated (*Zeitsch. Anal. Chem.*, vol. xxi., p. 33) that hardly any chemical data existed for the examination of distilled beverages, fair progress has been made in the working out of methods for detecting and determining the amounts of the various *classes* of substances above enumerated, although, as we all know, much remains to be done. But no advance worth recording has been made—nay, barely has the attempt been made—to differentiate between the individual members which make up each class of these "impurities." Nor is there much hope that such differentiation of closely-allied substances, the sum total of which amounts to almost intangible traces, will be practicable for the purposes of the Public Analyst. Let us take only the "higher alcohols," or "fusel oils," which generally form the larger proportion of the "impurities." It is known that the higher the molecular weight of an alcohol, the greater its toxicity. It is also known that secondary alcohols are more noxious than primary and unsaturated more than saturated; yet the analyst must content himself with determining, as best he may, the sum total of the higher alcohols, leaving altogether aside the question whether these consist of harmless or noxious members of the group. Again, the analyst can, with satisfactory accuracy, determine the amount of alkali required to hydrolyse those constituents which are called "esters," and to calculate the alkali consumed into any imaginary member of the "ester" group; but whether the constituents thus measured consist of valueless ethyl acetate or of complex and valuable members is beyond his ken; and so on with each one of the groups.

Analytically, therefore, the fringe only of the subject has been touched. A vast mass of analytical data, valuable as far as they go, has, however, gradually accumulated. Hundreds of analyses of brandy, and of other similar beverages, have been published during the last ten or more years, mainly by Girard and Cuniasse, Rocques, Sell, Mastbaum, Allen, Schidrowitz, and many others; and pages of figures containing analyses as full as practicable—quite as full as the few of the *Lancet* Commissioner (some of which are quoted from French sources)—are contained in Koenig's magnificent collection ("Chemie der Menschlichen Nahrungs und Genussmittel," fourth edition). New series of analyses are continually being published, the latest being an admirable contribution by Karl Windisch (*Zeitsch. Unters. Nahrungs und Genussmittel*, vol. viii., October, 1904, pp. 465-505). This closely-reasoned paper is worthy of very attentive study. Among other things, Windisch disposes once more of the suggestion which has been made by Loock (*Zeitsch. offentl. Chem.*, 1900, vol. vi., p. 397) that all genuine wine distillates contain furfural, whilst silent spirits were said to be free from it. Furfural is shown to be an accidental product of the decomposition of the pentoses; it is often—but by no means always—present in brandies. As a measure of genuineness it is useless. Windisch further discusses the much-debated claims made by Girard and Cuniasse, Rocques, and other French chemists, for the "coefficient of impurities," the sum total of volatile acids, esters, aldehydes, higher alcohols, and furfural, expressed in parts per 100,000 of the water-free sample. The coefficient of impurity for real cognac is alleged to be never less than 300, according to Girard and Cuniasse, and to Lusson not less than 340, while Rocques further claims that pure wine distillate contains approximately equal quantities of higher alcohols and of esters. As regards the latter relation, I may at once point out that in Rocques, "Analyse des Alcools et des Eaux de Vie," analyses of real cognacs are given showing this proportion to vary from 0·9 to 2·5 (higher alcohols divided by esters), while in other real brandies—not cognacs—the proportion varies from 0·43 to 2·92. As regards the "co-efficient of impurities," it may be worth while to quote the averages of all analyses of Girard and Cuniasse, as given by Koenig (*loc. cit.*), p. 1511, etc.:

Industrial alcohols ...	17·6
Real cognacs ...	486·3
Normal cognacs ...	473·3
Commercial cognacs (known brands) ...	257·3
Artificial cognacs ...	57·8
Marc brandies ...	852·1
„ „ (fair quality) ...	238·5
Kirsch brandies ...	401·5
„ „ ...	448·6
„ (imitation) ...	64·6

Rum	690·5
,,	593·9
Mixtures	299·4
,, (artificial)	104·6
Cider brandy	710·2
Whisky	409·4

The figures for "real cognacs" vary from 259·6 (a sample marked "Aunis, good, but not warranted") to 1174·3 (a very old cognac), while the "commercial cognacs" show variations from 74·6 to 703·2. It may be that the "commercial cognacs" include admixtures; but there is no evidence to that effect. A sample, twenty years in bottle, and therefore presumably genuine, showed a coefficient of "impurity" of 131·7. The limit of 300 cannot, therefore, be maintained, even for the cognacs analysed by MM. Girard and Cuniasse. In an article like brandy it is practically impossible to furnish unexceptionable evidence of its purity. No single person would ever be in a position to follow its production from the vineyard to the bottle. Therefore proof of genuineness cannot easily be obtained. On the other hand, the classification into "real cognacs," "normal cognacs," "commercial cognacs," and "artificial cognacs," adopted by Girard and Cuniasse, is altogether arbitrary, and as a rule based upon nothing better than the figures obtained by analysis, and as the fixation of a limit of "impurities" is an arbitrary process, this procedure begs the whole question. Similarly, referring to Mr. Vasey's book on "Analysis of Potable Spirits," the classification into types of brandies (which ought really to be types of cognacs) and types of blended spirits rests upon insufficient evidence.

For brandies of sources other than those of the cognac district, it has been conclusively proved upon evidence which is exceedingly strong that the coefficient of "impurity" is quite inapplicable. Thus Mastbaum (*Zeitsch. Unters. Nahrungs und Genussmittel*, 1903, pp. 56, 57), analysing and recording the results of twenty Portuguese brandies of authenticated origin, and following Girard's methods of examination, finds the "coefficient of impurity" to vary from 107·5 to 977. In nine samples out of twenty it was less than 300, while the relation of esters to higher alcohols, instead of being unity or thereabouts, varied from nine to one. Mastbaum also finds furfural absent in some of these samples, among these the very finest.

The "coefficient of impurity" has now, however, officially received its quietus, a report by a committee appointed by the French Minister of Commerce by a decree of March 22, 1904 (which report was published in the *Moniteur Officiel de Commerce* of June 30, 1904, and is commented upon in *Nature* of November 3, 1904) showing that while ordinarily the coefficient in cognacs and *fine champagne* ranges between 275 and 450, it is less in Armagnac brandies, while in those from the Midi and Algeria it varies from 25 to 500.

What possible value can attach to an analytical factor which varies from 25 to nearly 1,200?

The French Committee, therefore, "are unable to recommend that any limit should be fixed," and they, as other observers before them, reported that "expert tasting (*dégustation*) must be considered as an indispensable complement of chemical analysis."

The slightest consideration shows that immense variations in the "coefficient of impurities" are bound to occur. By keeping, large proportions of aldehydes are formed; when the brandy is made, as it often is, from wine that is more or less "off," variable amounts of acetic acid pass into the distillate, and the acidity, again, controls the amount of esterification. As regards the higher alcohols, which mostly form a very large proportion of the total "impurities," these are for the most part not products of the alcoholic fermentation by yeasts at all, but are formed after the chief fermentation is over by bacterial action upon carbohydrates (Emmerling, *Berichte*, 1904, p. 3535), under anaerobic conditions, and their production is not under practical control of the vintner. But the manner of distillation more than anything else controls, and is bound to control, the composition of the distillate. If from a badly-fermented wine the spirit be distilled off by crude means, large quantities of fusel oil and other products pass over—little from sound wines distilled, as they sometimes are in distilleries, from the water or the brine bath. The first fractions of the distillate are naturally comparatively rich in "impurities." Experiments made by the Swiss Government show that when wine is distilled the fusel oil coefficient varies from 660 to 170 in successive fractions. The *Lancet* commission's table on p. 1511 exhibits variations from 334 to 37. Taking all circumstances into consideration, it follows that the "coefficient of impurities" is largely an accidental quantity, applicable, under proper restrictions, to products made in the same way from approximately uniform crude products, but not applicable to all brandies alike, nor useful for the purpose of fixing a standard under the Sale of Food and Drugs Act.

The French originators of the coefficient knew this full well, for, with all their advanced knowledge of the present time, they are in the same position as were Nessler and Barth in 1883, when they said (*loc. cit.*): "Without doubt the sense of taste and smell of the connoisseur are better reagents for quality and purity of brandy than are chemical means," a position confirmed by W. Fresenius, 1890 (*Zeitsch. anal. Chem.*, p. 283); again by Sell in 1892 (*Arbeiten a.d. Kaiserl. Gesundheitsamte*, vol. vi., p. 335), who warns the analyst that he cannot analytically prove with certainty the purity or impurity of any sample, but that he has also to consult his senses; by Bersch (*Nahrungs. Genussmittel*, vol. ix., 1895, p. 116), who insists upon the same thing, and suggests that in every case involving a prosecution the judgment of the chemist must be confirmed by that of one or more tasting experts. In 1895 Rocques (*Moniteur Scientif.*, April) strongly recommends to combine chemical analysis with *dégustation*—that is, tasting and smelling the brandy in its concentrated form, as well as diluted with warm water. Girard and Cuniasse recommend the same course, and Koenig, summing up in 1904, with the knowledge of all the analytical work published up to that time, warns the chemist that "taste and smell by experts afford a safer foundation for an opinion than does chemical analysis."

We judge nearly all our food-stuffs, certainly all our "Genussmittel," as distinguished from "Nahrungsmittel," by our senses of taste and smell, and not by analysis. We cannot distinguish the cheapest from the most expensive wine by analysis; I know of no analytical difference between a halfpenny and a one-shilling cigar, nor between the cheapest tea and the finest. Flavours have been and are still, in most cases, beyond our analytical grasp.

Having quoted the opinion of the above chemists who have specially concerned themselves with the analysis of brandy and other alcoholic distilled liquors, I would contrast with it, without further comment, the position taken up by some Public Analysts in this country, namely, that the determination of the amount of ethers is sufficient to form a judgment upon which a vendor may be criminally prosecuted—nay, further, that this single determination justifies them in giving a definite certificate to the effect that a sample, because deficient in ethers—judged by an imaginary limit—contains a definitely-stated percentage of spirit *not derived from the grape.*

It would be easy to quote from the hundreds of published brandy analyses numbers referring to ethers only, and which show that these fluctuate as widely almost as the "coefficient of impurities." In undoubtedly genuine brandies ether coefficients have been found as low as 18 and as high as 450. I myself have had in hand quite a number of brandies which I have received from responsible persons, and with every assurance that they consisted of nothing but good commercial wine distillates, and which yet contained far less than the limit of 80, which is associated with certain recent legal proceedings. Almost in every instance when a brandy not produced in France is examined the ether number is low, and to say that a producer of a wine distillate in Algeria, Spain, Portugal, California, or Australia, has no right to call his distillate a "brandy" would be as unreasonable as to suggest that the Russian peasant has no right to call the fat which he churns from his milk a butter, simply because it sometimes differs in composition from that of the Australian or English producer.

This position is emphasized by the necessity under which some analysts have lately found themselves of adopting as a definition of brandy "an alcoholic liquid obtained from wine by distillation in *pot-stills.*" It may well be that a wine distillate from "pot-stills" has a stronger and perhaps more valuable flavour than a product made from some other kind of still; but this, I maintain, is a question for the fancy of the consumer.

It is interesting to inquire what is the probable reason for the selection of the "ethers" out of the whole list of concomitants which chemists have found necessary to determine for forming the best possible judgment on a "spirit."

I am firmly convinced that this selection is due mainly to prejudice. The chemist associates the term "ethers" with pleasant-smelling, aromatic substances. That the "ethers" contribute their quota towards the flavour of a brandy must be obvious; but that they are far removed from being the sole, or even the main, factors is apparent from the fact than when an alkaline hydroxide is allowed to act in the cold upon a brandy the aroma undergoes comparatively little change. For all other constituents which the analyst can determine, no good case in favour of brandy can possibly be made out. In the eyes of the public, the Bench, and especially in those of the Medical Profession, it would not be considered a recommendation if they were told that brandy, as a rule, was exceptionally rich in fusel oil, for fusel oil has for many years past been associated in the public mind with all that is undesirable in spirituous liquors. Nor would it help to call in the assistance of furfural, which is poisonous, nor of aldehydes, which are irritating and obnoxious. As brandy, however, doubtless occupies a high position in the public opinion, and as the main impurities present in it cannot possibly be considered in any favourable light, especially from a medical point of view, it is but a small step to attribute to the ethers virtues which they probably do not possess, or for the possession of which not a particle of scientific evidence has ever been brought forward. In the various cases which have been before the police-courts of late, an important feature of the prosecutions was the evidence of medical men who spoke eloquently about the medicinal properties of brandy, which were alleged to reside in their ethers. As a matter of fact, all unrefined spirits, no matter from what source, are comparatively rich in ethers. The difference in the ether content of brandy and of whisky is often insignificant, whilst the quantity in rum is frequently very high. It is highest of all in the very commonest of brandies made from the residues, the skins and pips of the grapes from which the valuable juice has been expressed. In spite of medical opinion, there is no doubt, to my mind, that the effects of a spirituous liquid reside in its alcohol, and for all practical purposes in nothing else, and that the function of the "impurities" is solely to give to the otherwise flavourless alcohol that zest which human beings require in all they eat or drink. It is quite well known that the best and most nutritious of diets is not tolerated unless it is properly flavoured. Dietaries that have been constructed with a disregard of that fact have led before now to catastrophes among prisoners and soldiers, and culminated in deaths. As a machine will not run without lubricant, although the latter adds nothing to the power, so the flavour of food or drink facilitates the work of the animal machine. The consumer must "like" his food for it to agree with him. The one may prefer the flavour of whisky, the other of brandy, as regards his beverage, but a chemical and scientific explanation cannot be given.

For years the impression has prevailed that brandy was a more healthful beverage than any other spirituous liquid on account of its erroneously presumed purity. We now know that, if anything, brandy is an impure material. There was a time when physiologists believed they could calculate and express numerically the toxicity of an alcoholic beverage, the amounts of aldehyde, furfural, some of the esters, and some of the higher alcohols necessary to kill an animal having been determined and calculated into kilogramme-factors by Dujardin-Beaumetz, Audigé, Rabuteau, and others; thus the quantities expressed in grammes necessary to kill 1 kilogramme of animal amount for ethyl aldehyde to 1·00 gramme, for acetic ether 4·00 grammes, for "higher alcohols" 1·40 grammes, and for furfural 0·24 gramme. Mastbaum, calculating the toxicity for a comparatively pure refined wine alcohol, with the "coefficient of impurity" of 175·7, and for a very impure brandy with the coefficient of 977·2, concluded that the "impurities" contained in 100 c.c. of these ("anhydrous") beverages would respectively kill 63·7 and 342·0 grammes of animal. This is disregarding the toxicity of the ethyl alcohol itself. According to Dujardin-Beaumetz and Audigé, this is 7·5, and 100 grammes of alcohol kill 12·9 kilogrammes. When this is allowed for, the toxicity of the two samples in question comes to 12,963 and 13,242 respectively—that is to say, the difference is altogether immaterial. It is clear that the toxicity of an alcoholic liquor belongs to the alcohol, and practically to nothing else.

We cannot escape the logical conclusion which was arrived at some years ago by the Departmental Committee on "Whisky," that pure alcohol—that is to say, silent spirit—is, if anything, a more healthful beverage than the ordinary drinking spirits—*all barring the necessary flavour.* Everything turns upon the latter. The importance which the consumer attaches to the origin of a spirit is infinitely less than that he attaches to the flavour. I am quite aware that I am uttering an unpopular heresy, but the clear outcome of recent analytical work with spirits proves up to the hilt that, broadly, the best-flavoured and the most valuable spirits are the least pure. Nevertheless, it may be true that the cheapest spirits are the most noxious, not because they are the most impure, for the purest spirit (silent spirit) is the cheapest of all, but just because for a given sum of money a larger amount of poison—that is to say, of alcohol—is obtainable in the one case than in the other. A gallon of "silent spirit" without the duty costs but a few pence, and cognac brandy as many pounds. No wonder that a given money value of the former can prove a poison where the latter imparts but the flavour to a cup of coffee. The subject is surrounded by a nimbus of popular prejudice, and it is comparatively easy to convince a magisterial bench that the vendor who supplies "silent spirit," which, largely because it is foreign and cheap, is credited with poisonous properties, injures the health of the community. Nevertheless, it is clearly against the spirit of our legislation to substitute one thing for another. If the health of the purchaser is not prejudiced, it may be that his pocket is. Of few things, however, the ordinary person is more capable of forming a correct judgment than of his drink, and we Public Analysts, I fear, can do but little at present to assist him in forming a judgment as regards derivation and quality. By the various prosecutions we have not only not assisted the purchaser, but an effect quite contrary to that intended has been produced. Just as certain regulations on the Continent have led to the appearance of a multitude of brandy-flavouring preparations, the analyses of scores of which have been published in Koenig's book, so a brandy-ether industry has commenced to flourish in England. I have it on the authority of one of the manufacturers himself. I also know from a large "brandy" merchant that so-called very cheap brandy is now sold with a guaranteed content of what they call "80 per cent." of ethers.

In view of the facts set forth above, it behoves Public Analysts to be cautious. It is their function to protect the public from fraud and to insure that the purchaser gets what he has a right to expect; but they must not go one tittle outside their knowledge of the facts. As scientific advisers, there must on no account be any pretence that something can be done by analysis which is beyond the chemist's knowledge. There is, it seems to me, some danger that zeal in the public interest may lead some analysts to take that course.

The names of articles of food and drink do not necessarily apply now to the selfsame substances as formerly. Beer was undoubtedly for a long time a malt and hop beverage; whisky, equally without doubt, was a product of fermented malt-wort. They are not so now. The product may be the same, or may be deemed by the public to be the same, but the raw material has changed. The time may come (I personally rather think that it will come) when brandy will mean only a brandy-flavoured alcohol, no matter from what source that alcohol is derived, just as the term "whisky" at the present time is legitimately applied to a whisky-flavoured fluid not necessarily made from malt. Public Analysts, by premature action based upon imperfect knowledge, are much more likely to hasten the advent of that time than to prevent it.

Discussion.

The President (Mr. Fairley) said he was sure they were all indebted to Mr. Hehner, whether they agreed with the views he had formulated or not, for the great care and trouble which he had taken in preparing this paper. He himself had not had any samples of brandy submitted to him for this purpose, and formerly he had tested the alcoholic strength and general characters of the distillate and the amount and nature of the fixed residue. As he had not had any samples, he thought he might claim to take up a more neutral position and to speak somewhat as an outsider. The Sale of Food and Drugs Act spoke of "the nature and quality of substances demanded" and of "prejudice to the purchaser," which meant that the purchaser should get what he reasonably expected to get. The ordinary purchaser had little knowledge of many of the articles he consumed. His idea might be founded on prejudice or on bygone experience, but he (the President) thought that when all was said and done, whether or not they adopted the views of Mr. Hehner, the purchaser, no matter how ignorant he might be, had some right to get what he expected to get. Mr. Hehner admitted that the time was when all brandy sold in this country was cognac brandy. With the extension of commerce and of the cultivation of the grape in various countries, and with the improvements in the methods of fermentation and distillation, came a variety of liquids whose status had now to be decided. Some of these points were defined in Circular No. 18 recently issued by the United States Department of Agriculture, drawn up by Dr. H. W. Wiley, the Chief of the Bureau of Chemistry of the Department. Brandy (potable brandy) is the distillate from wine, properly aged by storage in wood to eliminate the greater part of the fusel oils, etc., which may be present. Brandy should not contain less than 45 nor more than 55 volume per cent. of alcohol, and not more than 0·25 per cent. of total solids (extract). The content of fusel oils should not exceed 0·25 per cent. Brandy should not be mixed with alcohol from any other source than that of distilled wine. The distillate from the lees, pomace, and refuse of the winery is not entitled to bear the term "brandy" in the potable sense. "Cognac" is only admitted as a name in the case of brandies made in Cognac from wines grown and manufactured there. No artificial colour other than that derived from the wood in which they are aged is admitted in brandies. One question was, had the Public Analysts who had brought this matter forward exceeded the reasonable limits of their duty? He knew of cases where very considerable pressure had been brought to bear on certain Public Analysts to go into this matter. He had very great sympathy with such cases and with those who were honestly trying to do their duty in dealing with the matter. Only by continuous effort could improvement be attained, as was well shown in the analysis of butter and even of milk.

Mr. Fisher said it had become necessary to form an opinion and take a line in this matter, and the responsibility was on him, as on others, of saying whether certain samples purchased under the Food and Drugs Acts were to be accepted as brandy. Some samples containing a very low proportion of acids, ethers, etc., were alcohol slightly coloured and flavoured, differing entirely from cognac, and had no title to be called brandy. Others had the flavour and odour of brandy, but showed a deficiency in certain constituents. Such samples were probably mixtures or blends of cognac with rectified spirit, which might have been made from grain, beetroot, or other material, and if the mixture could be proved, they were not genuine brandy. It could not be maintained that a grape brandy might be mixed with whisky or gin (if that were possible, apart from the flavour) and the blend sold as brandy. He must admit, as Mr. Hehner had stated, that it was not always possible to detect the origin of the alcohol when blended in the brandy; yet when alcohol had been produced by rectification from grape spirit it was a debatable point whether it became brandy by being mixed with another proportion of actual grape-brandy. He did not, however, believe that in nine-tenths of mixtures met with the alcohol was exclusively derived from wine. They were, in fact, compounded partly of what everybody would regard as brandy, the other spirit being what nobody would admit was brandy. Mr. Hehner's remarks indicated the need for careful examination of these cases, and assertions must not be made which could not be substantiated. He did not regard this as a question of the differences in composition shown by the varieties of brandy made in Algiers, Greece, Spain, or Australia. Each had, of course, its own characters. He had given his opinion on samples which were afterwards admitted to be mixtures, and he had examined samples which were labelled and sold as mixtures. With reasonable care and precaution, he thought it possible to certify that a sample was genuine grape brandy or that it was a blend of the kind described.

Mr. A. H. M. MUTER thought that it would be a great pity if the Society of Public Analysts came to the conclusion that they were unable to deal with this question. He thought it would be far better that an effort should be made and a standard fixed, as had been done in the case of milk.

Mr. GORDON-SALAMON said that they must start first of all with the question which was the subject of the paper, namely, What is brandy? He thought the fact had to be accepted that the public regarded brandy as spirit derived from the grape, and in giving evidence he should certainly support that view and no other. If he were correct in assuming that position, then the question for the analyst to determine was whether by analytical methods he could say "Aye" or "Nay," is the public getting that article? Arising out of it was the further question—Does the present state of our knowledge permit us to come to a definite and reliable decision in respect of such a question? With all respect to the President, he (Mr. Salamon) did not agree with the view expressed, which in effect said: "Do not let us be discouraged. If we have not yet got to perfection of knowledge, we shall later." But meanwhile vendors were being prosecuted, and evidence was being given by public analysts to the effect that such and such samples were adulterated with spirit not derived from the grape, when at that meeting members were expressing grave doubts as to their being justified in making such a statement. In his opinion, that was a position which did not reflect credit on the Society, and until they were provided with some definite and proper method of analysis (which was approved by the profession as a whole) they ought to be extremely careful in making statements upon oath before magistrates, who would naturally look to them for guidance. On the other hand, one had to examine into the origin of this question. Mr. Hehner had very properly mentioned that it was due to the scarcity of brandy during the phylloxera ravages, and those who had investigated the manufacture of brandy, as he (Mr. Salamon) might claim to have done, in some at least of the countries of its manufacture, knew that adulteration with grain spirit was very largely practised. He knew as a matter of fact that very large quantities of grain spirit had in the past been employed for the adulteration of brandy. He thought the practice was reprehensible, and should, if possible, be stopped. Therefore, he was one of the first to admit, as the result of his own personal knowledge, that brandy was subject to adulteration with grain spirit, and that the public very often did not get what they demanded in the shape of brandy. But that being admitted, the next question was, Could it be determined by analysis whether or no such addition had been made? Now, what was the condition of the grain spirit as usually employed? It was nearly always pure rectified spirit—as pure a spirit as could be found—and purchasers were extremely careful in their choice of such spirit. Assuming also that brandy was to be derived from the grape, was it justifiable to add distilled grape spirit to that brandy, or must brandy be absolutely the untreated product of distillation? Was it allowable to add spirit derived from the grape in the pure state at a later stage? In answering that question one must not forget that it was the common practice of the French brandy manufacturer to add pure spirit at a later stage, and to the bulk of the brandy sent from France pure spirit was frequently added, and often in very considerable quantity. Therefore, in seeking to determine the origin of the spirit and the terms of its purity, could an analyst say "Aye" or "Nay," as to whether that spirit—assuming it to be rectified spirit—had been derived from the grape or from grain, or from any other source? Mr. Fisher had clearly answered that question, and he (Mr. Salamon) held exactly the same view. It was absolutely impossible to make such an assertion. If that were the view of that meeting, if that were the view of public analysts as a whole (and he did not see how they were to come to any other conclusion), what became of the whole question? Assuming a spirit—a brandy—containing 150 parts of esters per 100,000 of absolute alcohol were in the course of manufacture diluted down with rectified spirit until the esters were below the stupid standard of 80 by perhaps 5, who could say whether that had been legitimately brought about by the addition of pure grape spirit or whether it was due to the addition of grain spirit? And if that could not be determined, how could it be said whether the brandy had or had not been derived from the grape. If one denied that issue, one was thrown back upon the one which did not obtain in practice—namely, that no addition must be made to the brandy after distillation. The articles published in the *Lancet*, if he might venture to say so, did not reflect credit on the leading medical journal of this country, because the expert who made the investigation, competent as he was in many respects, had confined his visit to Cognac, and the report of the so-called Commission favoured the impression that it dealt with the whole question of brandy upon a broad basis. He (Mr. Salamon) did not think the expert knew of the existence in France of many thousands of *bouilleurs de cru*, small farmers who were permitted by special legislation to make brandy from their own cultivated grapes, if they so desired. They were allowed to distil the spirit from their grapes instead of having to put an inferior wine upon the market. That permit had been inaugurated by the Government. Such brandies were sold, and many of them went into the Cognac district itself, and did so to this day, although the law had been altered in respect of their production. On analysis, such brandies, having been distilled, not in pot stills, but in nearly all cases by various modifications of the patent still, showed a great range of composition, which was absolutely different from the composition of pot-still brandy as it was made in Cognac. Yet those brandies were genuine spirits derived from the grape. Spanish, Algerian, and Australian brandies were absolutely in the same category, and only a relatively small proportion of the total brandy produced came from Cognac itself. Consequently, it was unfair to set up a standard, even assuming it could be supported by analysis, which only applied to one portion of the brandy-producing area, and that by no means the largest. Supposing, however, this standard were accepted, would it not result in opening the door to sophistication? What easier than to construct a brandy that should answer the standard requirements of the magistrate—to make it up to contain the requisite proportions of ethers, furfurol, aldehydes, etc.—and then send it out under the certificate of the public analysts of this country as a pure article? Mr. Hehner had said that a spirit might be judged by taste as well as, or even better than, by analysis; but he (Mr. Salamon) thought that they must be very careful in making such a statement. It might certainly be left to the taster to say whether the public would accept a brandy, and how much they would pay for it; but he (Mr. Salamon) did not think it could reasonably be left to him to say whether a brandy had been broken down with rectified potato, grain or grape spirit.

Mr. C. G. Moor said that he spoke with a certain amount of trepidation, because his views were to a large extent opposed to those of Mr. Hehner, who had considerable advantage over him in point of knowledge of the literature of the subject. He thought everyone would agree that brandy had, in the past at any rate, possessed a considerable reputation as a medicine. It was referred to in the British and many of the other present Pharmacopœias, and was in some of the earlier editions also. It was in the American Pharmacopœia, which gave a long list of tests to provide against sophistication; and it would also be found in the Austrian, German, and Hungarian Pharmacopœias, in the Dutch Supplement, and in the Swiss Pharmacopœia. The making up or "faking" of brandies by colouring silent spirit was certainly regarded as an adulteration in Canada. The Government Analyst there, in one of his bulletins, stated that the existence of this manufacture had long been known, but that it was only recently that the various ethers, etc., had been offered publicly for sale. Regarding the value of the ethers and their interpretation, the view which he (Mr. Moor) held was that the 80 parts of ethers per 100,000 of alcohol was simply a working standard, just as long ago 2·5 per cent., and then 2·75 per cent., and now 3 per cent., was a working standard for fat in milk. Even now it was perfectly well known that a great deal of milk was "let down" to the 3 per cent. limit, but, nevertheless, it did provide against, and formed a working basis for detecting, the worst adulteration. It was in this way that he thought they should do what they could in regard to brandy, rather than follow what might rightly be described as a policy of despair. He thought, therefore, that they must have some kind of standard, and that the 80 parts of ethers per 100,000 might be used for the moment. He ventured to think that in the case of brandy of any well-known make a sample manufactured before this agitation arose would contain over 100 parts of ethers per 100,000. It was perfectly clear, of course, that ethers could be added to silent spirit suitably coloured, and that such an article would pass the suggested standard as regards ethers; but it was quite obvious that samples containing as little as 20, 30, or 50 parts of ethers per 100,000, were not genuine brandy. He regarded brandy as the spirit derived from the grape in a pot-still, simply because that was the origin of brandy before modern methods were introduced. There was one condition, however, under which the proportion of ethers might fall below an arbitrary standard. When brandy was made from "sick" wines it was customary, he believed, to throw away the first part of the distillate, because it was nasty and could not be mixed with the rest, and because, the distillation being carried out in a country where there was no alcohol regulation, it did not matter about a portion of the distillate being thrown away. About a month previously he had been permitted to read a paper on this subject at a meeting of the Therapeutical Society, at which were present several of the most prominent medical men of the day; and he had particularly invited an expression of opinion as to whether there was really any physiological action or advantage in a good genuine brandy, or in a good wine or whisky, apart from the alcohol present. He had been surprised to find that none of the medical men would specially advocate brandy, or say that there was any advantage in having matured spirit. The question from the point of view of the Public Analyst seemed perfectly clear. The purchaser was entitled to be supplied with what he asked for, and anyone, whether educated or ignorant, wanted in the case of brandy what he believed to be the genuine article, and not artificially coloured silent spirit.

Mr. JOHN HERON said that probably an expert in spirits would be able to distinguish between wine-spirit and grain-spirit by some similar method to the one he had himself employed for many years for distinguishing between beet and cane sugar. There was a subtle something clinging to the refined, rectified article which stamped it at once as to its origin.

Mr. SALAMON: Whether it is pure or mixed with brandy?

Mr. HERON said that the difficulty that arose in the case of a mixture was that the constituents or impurities of the brandy would (and, indeed, must) belong to the same order or quality as those contained in the grain-spirit, so that it would probably be a difficult thing to attempt; but just as, in the case of sugars, the presence of an admixture (though not its quantity) could be detected, he should think it ought to be possible, after some experience, to tell whether grain-spirit had been added to brandy. He had hoped that at that meeting some understanding might be come to as to what ought to be the necessary constituents of brandy. However, anyone who had been experienced in the analysis of brandy and other spirits would, in the very handling of the liquid, come to know instinctively whether a particular sample was genuine brandy or not. He did not think that a right judgment could be formed on the basis of one constituent only, but by comparing the different constituents one with another a very fair opinion could be arrived at as to the quality of the brandy.

Mr. J. B. P. HARRISON said that the question of the sale of "British brandy" was an interesting one. Some Public Analysts held that there should be no such substance as "British brandy," as the term "brandy" in this relation was a misleading one; and as the chief consumers of this kind of spirit were not sufficiently educated to distinguish between it and actual brandy, the question was whether the purchaser knew what he was really receiving for the price he paid. He was also of opinion that so long as the Excise recognised the sale of spirit in the guise of "British brandy" as a legitimate source of revenue, there would always be confusion as to what was really genuine brandy.

Dr. SCHIDROWITZ said that he felt some little difficulty in speaking on this question from a purely analytical point of view, because, regarded in that light, it resolved itself into a series of metaphysical speculations. If he were to ask himself, —What is brandy from an analytical point of view?—he should reply that he did not know; and he thought they were all in that position. He did not quite agree with Mr. Hehner about the literature of this subject. He thought that the literature on the subject of brandy upon which one could actually pin any faith was very small indeed; and he did not agree that it was an impossible matter to practically substantiate the genuine character of a spirit. So far it had certainly not been done, and, whether one regarded the analyses of Girard and Cuniasse, of Mastbaum, or of any of the other observers mentioned, none of those analyses, as far as he knew, represented samples taken at the still-head. The grape, of course, could not be traced all through its growth up to the time when it was pressed and the brandy made from it, but there were practical conditions—it was not necessary to make a special plea in any direction —under which samples might be taken so as to enable it to be shown within what limits the different qualities and types of brandy did vary. That had not been done, and he must say that, until it had been, the industry and trade concerned had themselves largely to blame for the position in which they now are. He did not quite agree with Mr. Hehner's classification of the by-products. His view was that, in addition to the substances mentioned by Mr. Hehner, there were many others about which, generally speaking, little is at present known. In saying this he was not speaking merely as a matter of speculation, but from various facts which he had observed, and which led him to say that there certainly were such substances. Another question was—What is brandy, looking at it from the point of view of the manufacturer, and not from the point of view of the analyst? After all, the chemist should not consider himself merely as a person who put things into a test-tube and took them out again, but he could be, and was, an aid to great industries and trades; and therefore he thought that this matter should be looked at from the point of view of the industrialist also. If it were said that brandy was anything distilled from the grape, whatever the degree of rectification, he would put this case: Supposing grape spirit to be rectified to the point of absolute alcohol, and to be coloured with burnt sugar, bottled and sold——

Mr. CHAPMAN: People would not drink it.

Dr. SCHIDROWITZ, continuing, said that the question arose—Was such an article brandy or not? The purchaser said he did not think it was brandy; the analyst expressed an opinion, right or wrong; but, broadly speaking, they were all agreed that such an article was not brandy. Where, then, was the limit to be set? He was not going to express any opinion as to the type of still that should be used, or as to the degree of rectification that should take place, because the matter, regarded from a purely scientific point of view, was in its infancy. At the same time, he thought it a mistake to go to extremes in either direction. With regard to the toxicity of the higher alcohols, he might mention that the kilogramme factor for the alcohols in general, according to Dujardin-Beaumetz, varied from about 6 for ethylic alcohol to 1·5 for amylic alcohol. He must say that he was strongly opposed to judging any fermented or distilled liquid on a single analytical figure. He did not say that in years to come it might not be possible in many cases to form an opinion by purely analytical means as to the character of a spirit, but he ventured to think that it would never be possible to do so on the strength of a single analytical figure. The case of wine analysis might be instanced. Standards had been set up by the German Government on the strength of many thousands of analyses, and yet, since those standards had been set up, cases had occurred of genuine wines which were entirely outside the limits. He had met with German white wines which he knew to be absolutely genuine, but which, by the official methods, showed no tartaric acid at all. Some white wines, again, contained about twice as much glycerin as white wines of a similar class usually contained, the high content of glycerin being perfectly characteristic of the particular wine in question. Anyone relying on a single analytical factor might find himself in the position, with a sample yielding abnormal figures of that kind, of causing a prosecution when the article was perfectly genuine. He (Dr. Schidrowitz) thought that nothing further was needed to demonstrate the absurdity of proceeding in that way. He had seen many certificates in which samples were stated to be genuine brandy on the strength of the analytical figures. He had not been studying this matter for the last few months only, but had been working on spirits for a number of years, and would never give an opinion as to the genuineness of a spirit on analytical figures alone, though it might in some cases be possible to say that it was not genuine. But the sense of taste could be so trained as to work in association with analysis, and then very often the chemist could do something useful; and even at the present time he could do a great deal if he worked in conjunction with an expert taster. Moreover, he could assist the manufacturer in his processes of fermentation, distillation, etc., and he could assist the trade with regard to blending, fining, and various other questions. Finally, the chemist could act as a policeman, but he did not think the chemist should confine himself to that rôle entirely. If an article was not genuine they were all agreed that speedy steps should be taken, though what those steps should be was another matter. He must demur to the statement in the American circular which the President had quoted, that brandy should " be properly aged in order to remove fusel oil," because, whatever it was that disappeared in the maturation of spirits, it was, in his opinion, practically certain that it was not the higher alcohols. In his own experience he found that old spirits contained on the whole rather more fusel oil than new spirits, and, theoretically regarded, there was no reason why they should not. This statement, however, with regard to fusel oil had been handed down from generation to generation, until at last it had come to be believed. With regard to the medical opinions which had been given from time to time with regard to brandy, he had been trying to discover the record of any scientific physiological experiments on genuine brandy, whisky, or any other spirit, side by side with plain alcohol, but there were no such experiments; and even if a medical man recommended genuine brandy, he did not know what would happen if some other spirit were tried. He (Dr. Schidrowitz) agreed that the effect produced was mainly due to the alcohol, and to nothing else; but he held the view that, where special and beneficial physiological effects, which could not wholly be credited to the ethylic alcohol, had been produced, these were to be ascribed rather to the age of the spirit than to its original character.

Dr. **Thorne** said that the fact rather seemed to have been lost sight of in the course of the discussion that a standard had been more or less officially put forward, and that the number—80 parts of ethers per 100,000 of spirit—had become more or less fixed in people's minds. It seemed to him that, as a Society of Public Analysts, they ought carefully to consider whether they were justified in letting that stand for present use. Whether they might eventually come to the conclusion that brandies when properly made might be above such and such a standard was a point which might develop as time went on; but he thought it an undoubted fact that at the present time really pure brandies did not all contain ethers up to the standard of 80 parts per 100,000, and that therefore to make that one point an absolute standard was an injustice on the one hand and a great danger on the other. Although he was not personally connected either with public analysis or with the spirit trade, he happened to know that at that moment there was a large consignment of real Cognac spirit which had been in England for something like ten years or more, and which had been bought at a high price with the best expert authentication, but which on analysis showed only 67 parts of ethers per 100,000. That was a spirit which had cost originally certainly seven or eight times the price of many spirits that would be passed on the standard of 80, and was really a very fine Cognac. The danger, on the other hand, that imitation brandies might be made up was, of course, one to be fought against; but there was a very great danger that, if this one particular standard were fixed at a point which was higher than was fair, really good and sound brandy would be condemned, and there would be a tendency to add small quantities of ethers, not for the purpose of making really sophisticated brandy, but in order to bring real brandy into a safe position with regard to police-court proceedings. That was undoubtedly a serious danger, and he thought, therefore, that the Society ought to go so far as to take the position that at the present time the ether standard of 80 parts per 100,000 was not justifiable, and ought not to be maintained as an absolute legal test of good brandy.

Mr. **Bevan** said that he could confirm what Dr. Thorne had said, having had occasion to examine a sample of 1848 brandy which showed only 57 parts per 100,000 of ethers.

Mr. **Cribb** said that, with regard to the adoption of the standard of 80 parts per 100,000 for the ethers, that nobody intended that to be taken as a rigid standard, but simply as a basis for calculation. Nobody would dream of prosecuting for 67, for instance, or any figure nearly approaching 80. In all the cases that had been brought into court the ether figures had been very much lower—as far as we knew, never more than 40. In those cases in which he was concerned they were very much lower—some lower, even, than those yielded by samples sold as "British brandy." It must be borne in mind that 80 was very far from being the average figure. The average proportion of ethers shown in the analyses by Girard and Cuniasse, and also by Vasey, was something like 136, and if Marc brandies were included it was about 180.

Mr. **Hehner**: That was for cognac.

Mr. **Cribb**: That in France is the same thing as brandy in England.

Mr. HEHNER: May I say that "cognac" in French and in English is "cognac"; "brandy" in French is simply "eau de vie"?

Mr. CRIBB said that he had been in a great many hotels in France, and had never been asked to have anything but cognac. The term "eau de vie" was never once used in his hearing, and it was notorious and openly admitted that the stuff offered as "cognac" for use with coffee might be anything. He was quite sure that in France the word "cognac" was used in the loosest possible manner. If the average was as high as 136, it was quite obvious that the number of cases in which genuine brandy came much below 80 must be very few indeed, and, as a matter of fact, amongst all the analyses of Girard and Cuniasse and of Vasey there was only one. He should like to ask Mr. Hehner if he could tell him the number in the papers by Windisch and others to which he had referred.

Mr. SALAMON thought it might interest the meeting to hear that a couple of weeks previously he had had, from one of the largest importers of brandy into this country, two samples which he had to report upon as being below 80 in ethers, and therefore not capable of passing the magisterial standard. His clients had thereupon sent him a letter, accompanied by a report from the French Government authorities, stating that those brandies (as a special matter) had been prepared under Government supervision. A certificate of origin and a certificate of purity were also produced, in which it was declared that both brandies were pure grape brandy.

Mr. CIRBB said that he did not dispute for a moment that samples did come below 80, but the number was very few indeed.

Mr. HEHNER said that there were scores of cases. In a report made by the *Lancet* commissioner himself three samples of Algerian brandy were shown to contain respectively 62·73, 47·6, and 55·3 parts per 100,000 of ethers; and the report continued: "The figures coincide with those obtained with genuine, though somewhat young, brandies." These Algerian brandies consisted of spirit derived entirely from the grape. Thus, the same authority that had fixed the standard of 80 had also fixed the standard of 47.

Mr. CRIBB said he should like to mention that Algerian and Spanish and most other brandies grown out of France had a very different flavour from that of French brandy, and would hardly be recognised as brandy by regular drinkers of that spirit.

Mr. SALAMON said that the brandies he had spoken of came from France.

Dr. SCHIDROWITZ remarked that the "certificate of origin" applied to anything, whether absolute alcohol or brandy, that was distilled from the grape.

Mr. CRIBB said that a good deal had been heard about rectified grape spirit, but he had never seen any published analyses of it, and should be pleased if Mr. Hehner could refer him to any. He had been himself trying to obtain samples of it, but had only succeeded in getting one, which contained 67 parts of ethers per 100,000. Possibly that was abnormal; if not, it showed that the presence in a brandy of rectified spirit derived from the grape would not seriously affect the validity of the ether standard if judiciously employed. With regard to Mr. Hehner's remarks to the effect that the value of brandy and of all spirits consisted entirely in their flavour, and that no value in the present state of knowledge could be attached to any one of the analytical constituents, it seemed to him (Mr. Cribb) that, if they were to go merely by flavour, any concoction whatever, as long as it tasted right, would pass muster, no matter how it was made. That, however, was entirely contrary to the spirit in which they had hitherto carried out the requirements of the Sale of Food and Drugs Act. With regard to the article in the *Moniteur Officiel*, of course everything depended upon what one accepted as being genuine brandy, and it seemed to him that even in France the question must be a very difficult one unless the manufacture could be followed all through. One difficulty arose from the use there of portable stills, which were carried about the country to anyone who wanted to employ them. It was quite obvious that brandy made in that way might be subject to all sorts of admixture, for there could be very little control over it. He did not know how far an Inland Revenue officer went round with the stills, but from what he had heard he did not think that that was done. It had been remarked that the laying down of a standard had a most unfortunate effect in opening up the way for adulteration ; but surely the absence of any standard at all would do this to a much greater extent. With regard to the question of tasting, Mr. Hehner and Dr. Schidrowitz were apparently in conflict. In all the cases that had been at all fought the evidence of the taster had been called in, and had confirmed the opinion of the Analyst. He did not see how anybody at the present time could say that a sample was genuine, but in some cases it was possible to say that one was adulterated, and with greater confidence, of course, if the analytical evidence were supported by that of the palate.

Mr. CHAPMAN thought that this question emphasized in a very marked degree the necessity for the establishment of a Consultative Board or Court of Reference such as had been recommended by a Royal Commission and by a Select Committee, and more especially a Board constituted in the manner indicated by Dr. Thorpe in the Minority Memorandum which he had signed in connection with the Royal Commission on Arsenical Poisoning—viz., a body including not only scientific men, but also manufacturers and others possessing special technical knowledge. Not only was it unfair to the Public Analyst to compel him, as is very often the case, to set up standards for himself, but such a proceeding was extremely undesirable from every point of view ; and he hoped that before long this function would devolve upon a Government Department possessing the necessary facilities for obtaining such scientific and commercial information as might be necessary for the purpose. Mr. Hehner had in his opening remarks made a very clear and lucid statement ; and he (Mr. Chapman) cordially agreed with a great deal that Mr. Hehner had said. He would, however, like to take the opportunity of expressing his dissent from the view which had been put forward in some quarters that brandy might practically consist of anything which looked and tasted like brandy, and which was analytically indistinguishable from that spirit. He (Mr. Chapman) thought that was a position which many of them would hesitate to take up, for it was one which, if carried to its logical conclusion, would lead to such assertions as that a mixture of glycerides similar to that occurring in butter, and incapable of being distinguished from butter by chemical analysis, might, if it possessed the requisite flavour, be sold as butter without prejudice to the purchaser. He felt that was an extremely dangerous position to adopt. The main point in connection with that meeting centred very largely in the answer to the question, Were they in a position to make statements such as were being made and sworn to in courts of law in reference to samples of brandy examined under a Criminal Act? So far as he was able to judge, he thought that the general feeling of that meeting was that they were not justified in making such statements. He thought there could be little doubt that the demand for ethers was very largely due to their supposed stimulating and medicinal effect. He was certainly not prepared to say that the ethers did not possess such valuable properties, but he did feel that it was an absurdity to demand that a spirit, the great bulk of which was used as a beverage, should conform to a certain analytical standard merely because a certain small proportion was used for medicinal purposes. It was for the Pharmacopœia Committee of the General Medical Council to see if they thought proper that brandy sold as a drug should conform to some required standard; but for the rest the purchaser must, and would eventually, be the judge as to whether what was supplied to him was good value or not. If it should become possible to distinguish analytically between grape spirit and that which was not grape spirit, well and good; but to insist that all brandy should conform to one analytical standard was about as absurd as it would be to demand that all clarets should conform to the analytical type of a Château wine. Mr. Heron had raised an interesting point as to the possibility of distinguishing between alcohol from different sources. He (Mr. Chapman) believed it to be a fact that alcohol obtained from grain, even when very highly rectified, if added to wine, could be readily detected by the trained palate, whereas alcohol obtained from wine was capable, on the other hand—to use a wine-merchant's expression—of "marrying" or blending perfectly.

Mr. Cribb, in reference to what Mr. Chapman had said as to the ability of the taster to distinguish between grain spirit and grape spirit, said that he knew of a large London buyer of Spanish wines which had to be fortified by the addition of spirit who had inserted in his contracts with the foreign producers a special clause providing that no grain spirit should be added; and he professed to be able to tell at once by the taste whether grain spirit or grape spirit had been added.

Mr. Hehner, in reply, said that he considered it to be entirely within the right of the State or its representatives to say, as guardians of the public, "We will only permit a thing to be called brandy if it conforms with certain arbitrary regulations which we, the Government, have a right to lay down." The State could exclude, if it chose, anything, although it might be legitimately made, from a particular source, and could say that it should not—in England, at any rate—be called brandy. The State could say if it chose: "We insist upon nothing but a certain quantity of ethers." But he (Mr. Hehner) thought that it was not the business of the analyst to do so. The analyst had absolutely no right to do so, and only imposed upon the magisterial Bench by suggesting that he could do such a thing. There were, he reaffirmed, a very large number of analyses on record—well-authenticated analyses, made by competent people—which showed this. He quite agreed that those analyses were not all made upon the same standard, for it was notorious that the methods of determination, especially in the case of the higher alcohols, gave widely varying results. But the figures obtained by each method were more or less comparable among themselves, and if that condition were accepted it might be said that there were many hundreds of analyses on record, though, if the matter were regarded in the strict, scientific sense, the number of trustworthy analyses was very small. There were very many cases on record in which brandy fell, as regards ethers, below brandy of authenticated origin, and he agreed that this happened with much greater frequency in the case of brandies not of French origin. But these were not chemical curiosities, but articles of trade that were imported to the extent of tens of thousands of barrels, and when an article was once imported, the public analyst, at any rate, had no means of ascertaining whether it came from one country or another. When all these things were taken into consideration, it seemed to be absolutely absurd to judge by Cognac standards an article which was simply called "brandy," just as it would be absurd to judge all wines by the higher German standard, or to use in any other case a measure which was not applicable. He did not advocate a policy of despair, but he did not want analysts to bolster themselves up with fairy tales, and to say that which they did not at present know, but which they might know at some future time. However far one went, it would not be found that alcohol, from whatever source, was anything different from C_2H_6O. While, however, impurities attached to the alcohol, he fully believed that it would be possible, more or less, to distinguish by taste—not because the alcohol was different, but because the impurities were still there. That was the sense in which he had spoken when he said that one alcohol was as good as another. He quite agreed that sugar from the beet and sugar from the cane could be distinguished, even if highly refined, by the traces of the mother liquors which they contained; but it was not suggested that cane-sugar was different from beet sugar. A trade expert was perfectly entitled to go into court and express his opinion, which might be a perfectly correct one, but he (Mr. Hehner) thought that an analyst had no right to make a statement which was based on taste. His function was to analyse the article, and to judge it from the chemical and physical point of view. He (Mr. Hehner) fully agreed that the analyst was in very many cases quite right when, having found that there was a deficiency of ethers, he gave his opinion that the article was not ordinary brandy, because undoubtedly very many artificial mixtures were made. Such mixtures were often sold as "British brandy" in the first instance, and afterwards sold by the publican without the qualification "British." The analyst, however, could not possibly in such a case say for certain that the deficiency in ethers was due to any artificial addition; it might be due to natural causes, and he (Mr. Hehner) did not think it was fair that they as public analysts should take upon themselves to make a definite accusation when the variation was just as likely to be due to other causes. If anyone had actually seen anything added, he was, of course, at liberty to say so, but that was a matter for the policeman rather than for the analyst. The question had been raised with regard to other articles—the case of foreign meat and English meat for instance—and he could not see why the same principle which applied in that case should not be applied to brandy or whisky. It was not weakness to say that there was no scientific means of distinguishing; the harm was done by pretending to do that which could not be done. That change which had been experienced in all other departments of the spirit industry must, he thought, irresistibly come to be applied in time to brandy. He perfectly agreed with Mr. Fisher that nine-tenths of these deficient brandies were mixtures that had been artificially "let down," but he was perfectly convinced that there was not only no one single determination that would be of any assistance in distinguishing such mixtures, but that no possible combination of the determinations now available would be of any assistance either. There were undoubtedly genuine brandies which were high in ethers and low in higher alcohols and *vice versâ;* and there were undoubtedly genuine brandies which were very high in aldehydes and low in higher alcohols. In fact, all possible combinations existed in undoubtedly genuine brandies. Why should they, who knew really nothing officially about the preparation of brandy, because it was not a British manufacture, pretend to know more than those whose life business it was to watch the production? No Government could have been more interested than the French to keep the brandy industry a national one, but they had failed, and had acknowledged it. Their last Report, which he had ventured to quote, was to the effect that it was impossible to lay down any standard, because the impurities varied from 15 to 1,200. If there were any Pharmacopœia which gave information as to how to ascertain whether its requirements were complied with, he should be very pleased to see it. It was very well to lay down certain laws, and to say that brandy should be so-and-so; but the public analyst had to say whether it was so, and the complaint against the British Pharmacopœia was just that it laid down a set of laws which no one could comply with or enforce because the analytical data were wanting. If a Pharmacopœia stated that, for its purpose, brandy should contain a certain quantity of ethers (200 parts per 100,000 if they liked), let it be so; but that, surely, could not be a question of brandy for the public. His own feeling was very strong on the matter. He confessed that not very long ago he should have taken a diametrically opposite position, and should have said that brandy must undoubtedly be made from wine. It might be that he was wrong in altering his view, but he found that neither he nor anybody else in this or any other country had been able to do any better—that there was no means of distinguishing, and that the more analyses that were added, the wider the limits became. That might be called a policy of despair, but the real reason was that they could not fight against Nature. With regard to the question of asking the Government to fix a limit, the Government had known for many years that adulterated brandy was imported, but had never raised its voice. Blending had taken place openly, and the public might have cried out, but the Government had done nothing. Why public analysts should take upon themselves to do that which the Government had refused to do, and which, in the case of whisky, they had deliberately avowed themselves unable to do, passed his comprehension.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Watered Milk by the Refractometer. A. E. Leach and H. C. Lythgoe. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1195-1203.)—Numerous tabulated results are given of determinations of the refractions shown by the sera obtained from genuine and watered milks. The instrument employed was the Zeiss immersion-refractometer (ANALYST, 1903, 91), but the Abbé refractometer may also be used. In no case did the serum of a pure milk give a lower reading on the Zeiss instrument than 39·0 at 20° C. (1·3424 Abbé), figures from 40 to 44 being usually obtained. A milk serum giving a reading of 42·40 when mixed with 10 per cent. of water gave a reading of 39·75, and with 20 per cent. of water, 36·90 (see ANALYST, 1903, 241). W. P. S.

A Thickening Material for Cream. F. Reiss. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 605-607.)—Attention is drawn to a solution, sold under the name of "Grossin," which consists of a solution of calcium oxide (5·50 per cent.) and sugar (10·50 per cent.). The mixture is opalescent, owing to the presence of a little calcium carbonate. It is intended to be added to thin or scalded creams to thicken them, and, at the same time, to neutralize the acidity of the cream.

W. P. S.

Detection of Artificial Colouring Matters in Pastry. K. Dannenberg. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 535-538.)—Wheat meal always yields some colouring matter when extracted with alcohol, and which cannot be removed by previous extraction with ether. The author therefore proposes to use 25 per cent. alcohol as a solvent, spirit of this strength dissolving neither the natural colouring substances of wheat nor of eggs. Thirty grammes of the powdered pastry are treated with 50 c.c. of 25 per cent. alcohol, and, after shaking, allowed to stand for six hours. If the alcoholic solution then be yellow, added artificial colours are present in the sample. In their absence the solution will be colourless or dirty gray. To prove the presence or absence of colours which are insoluble in alcohol, but soluble in ether, the sample is extracted several times with boiling absolute alcohol, and then treated with ether. Should the pastry not be quite discolourized by the alcohol, but completely so by the ether, an ether-soluble dye is present.

W. P. S.

Characters of Indian Beeswax. D. Hooper. (*Agricultural Ledger*, Calcutta, 1904, No. 7; through *Pharm. Journ.*, 1904, lxxiii., 505.)—The author furnishes in his paper a very complete account of the sources, preparation, and composition of British Indian beeswax. Derived from the three under-mentioned species of bees, the waxes, as separated from the combs, are practically identical in composition, and are rarely adulterated. The chief source is *Apis dorsata*: The wax differs from

European beeswax mainly in its lower acid value. The large quantity of the wax available renders it a probable and valuable commercial source of supply.

The waxes have the following analytical characters:

		Apis Dorsata.	*Apis Indica.*	*Apis Florea.*
Melting-point, °C.	maximum	67	64	68
	minimum	60	62	63
	mean	63·1	63·2	64·2
Acid value	maximum	10·2	8·8	8·9
	minimum	4·4	5·0	6·1
	mean	7·0	6·8	7·5
Ester value	maximum	97·8	95·9	123·8
	minimum	69·5	84·0	80·8
	mean	89·4	89·6	95·6
Iodine value	maximum	9·9	9·2	11·4
	minimum	4·4	5·3	6·0
	mean	6·7	7·4	8·0

Some wax is also collected from the allied Dammar or Kota bees, Melipona (Trigona) species. These very small stingless insects produced a sticky, dark-coloured wax, having a melting-point of 70·5° C.; acid value, 20·8; ester value, 89·6; and iodine value, 42·2. The product more nearly resembles the propolis of honey-bees than true wax, from which it differs largely in physical and chemical characters.

A. R. T.

Determination of Added Mustard Hulls in Ground Mustard. A. E. Leach. (*Jour. Amer. Chem. Soc.*, 1904, xxvi., 1209, 1210.)—Whilst the higher grades of mustard flour are almost entirely deprived of hulls, some are occasionally met with containing added hulls to an extent exceeding the amount of hulls in the whole seed. In the author's opinion this should be considered a form of adulteration. The main points of difference between hulls and seed substance are indicated chemically by such constants as the total nitrogen, crude fibre, and the copper-reducing matters by the diastase method. The following table, in which the results are given on the dried and fat-free substance, shows this in a summarized form:

		Mustard Flour.	Mustard Hulls.	Whole Seed.
Total nitrogen, per cent. ...	maximum	9·61	5·00	7·73
	minimum	8·27	3·40	6·00
	average	9·09	4·14	7·09
Crude fibre, per cent.... ...	maximum	4·26	22·20	10·33
	minimum	2·31	13·74	7·24
	average	3·24	18·11	8·05
Reducing matter (as dextrose)	maximum	0·93	7·06	3·13
	minimum	0·00	1·51	1·39
	average	0·37	4·27	2·40

The following limits are suggested for ground mustard, expressed on the dry, fat-free substance: The reducing matter (as dextrose) should not exceed 2·5 per cent., the crude fibre should not exceed 5 per cent., and the total nitrogen should not be less than 8 per cent. As shown by the microscope, the sample should be free from more than minute traces of starch, and should not exhibit an excess of hulls over seed tissue.

W. P. S.

Composition of Orange Juice. K. Farnsteiner and W. Stüber. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 603-605.)—The juices to which the following figures refer were pressed from the oranges after removing the skins, spongy tissue, and seeds from the latter. The press and vessels for receiving the juice consisted of wood, no metal being present.

Grammes per 100 c.c.	From Oranges of Unknown Origin.		From Valencia Oranges.	From Messina Oranges.
	Fresh.	Fermented.		
Specific gravity at 15° C.	1·0429	1·0084	1·0464	1·0451
,, after boiling	1·0454	1·0159	1·0466	1·0455
Total solids, dried two and a half hours in water-oven	10·73	3·55	10·92	10·85
Citric acid (anhydrous)	1·19	1·25	1·79	1·47
Total sugar, after inversion (as invert sugar)	8·26	0·38	7·65	7·86
Ash	0·41	0·42	0·52	0·50
Alkalinity of ash (c.c. of N acid) ...	5·40	5·62	7·20	6·40
Nitrogen	0·064	0·053	0·099	0·075
Phosphoric acid	—	0·026	0·027	0·042
Glycerol	(0·38)	(0·61)	(0·34)	(0·28)
Alcohol	1·44*	4·29	—	
Polarization in 200-millimetre tube, direct			- 0·11°	+ 2·45°
Polarization in 200-millimetre tube, after inversion			- 3·16°	- 3·66°

W. P. S.

Determination of Mustard Oil in Mustard Seeds. Vuillemin. (*Pharm. Centralh.*, 1904, xlv., 384; through *Schimmel and Co.'s Report*, October-November, 1904, 59.)—The author proposes the following modification of K. Dieterich's method: Five grammes of mustard seed are triturated as finely as possible with a little water, transferred to a round-bottomed flask of 200 c.c. capacity, and 100 c.c. of water at a temperature of 20° to 30° C. added. The mixture is then allowed to stand in the closed flask for one hour with frequent agitation. After the addition of 20 c.c. of alcohol, the flask is connected to a Liebig's condenser, an Erlenmeyer flask of 200 c.c. capacity, containing 30 c.c. of ammonia and 10 c.c. of alcohol, being used as the receiver. To this is connected a second flask, containing ammonia and alcohol, to avoid the possibility of loss. About half the contents of the distilling-flask are distilled over while the condenser-tube is immersed in the liquid in the receiver.

* A small quantity of an alcoholic solution of salicylic acid had been added to this juice.

When the distillation is at an end, the condenser is rinsed with a little water, and the distillate in the flask mixed with 3 to 4 c.c. of 10 per cent. silver nitrate solution, and heated on a water-bath until the silver sulphide precipitate is well deposited and the supernatant liquid is quite clear. The hot liquid is filtered on to a tared filter, the precipitate washed with a little hot water, then alcohol and ether, and dried at 100° C. until constant in weight. Any silver sulphide from the liquid in the second receiver must be added to the main quantity. The amount of silver sulphide obtained, multiplied by 8·602, indicates the percentage of mustard oil in the seeds when 5 grammes of the sample have been taken for the analysis. A. R. T.

A New Adulterant of Ipecacuanha. C. Mannich and W. Brandt. (*Journ. Pharm. Chim.* [6], xx., 276; through *Pharm. Journ.*, 1904, lxxiii., 705.)—The root of *Heteropteris pauciflora*, already mentioned by Peckolt, of Rio, as being used to adulterate ipecacuanha, has been examined by the authors. Whilst the exterior appearance of this root closely resembles that of ipecacuanha, its histological structure is markedly different. It contains no starch or raphides of calcium oxalate, but shows cells containing a peculiar brown colouring matter which is coloured black by ferric chloride; the structure of the woody tissue and of the more highly developed parenchyma also differs from the corresponding parts of true ipecacuanha. In place of starch there is present a carbohydrate soluble in water, which, after precipitation by alcohol from its aqueous solution, and redissolving in water, yields a clear syrup on evaporation. When purified by reprecipitation and solution, the carbohyrate has the formula $C_6H_{10}O_5\frac{1}{2}H_2O$ and $[\alpha]_D - 40.\ 98°$; it does not reduce Fehlings's solution, is precipitated by barium hydrate, and yields lævulose on hydrolysis. It is therefore allied to bodies of the inulin group, and has been provisionally named heteropterin. *Heteropteris* root contains no alkaloid, but yields a small amount of a crystalline non-glucosidal substance. W. P. S.

Colorimetric Valuation of Rhubarb. A. Tschirch. (*Pharm. Centralb*, xlv., 496; through *Pharm. Journ.*, 1904, lxxiii., 428.)—The author's method consists in the hydrolysis of the anthraglucosides of rhubarb, and the solubility of the resultant oxymethyl-anthraquinones in ether. Half a gramme of the finely powdered sample is boiled for fifteen minutes under a reflux condenser with 50 c.c. of dilute sulphuric acid (5 per cent.). The cooled liquid is directly extracted with successive quantities of ether until the solvent shows no colour on the addition of a trace of caustic potash. The mixed ethereal extracts are next evaporated, when the process of heating with dilute sulphuric acid and the subsequent extractions with ether are repeated on the residue. After complete extraction of the acid liquid, the ethereal extracts are mixed and shaken with successive quantities of caustic potash solution (5 per cent.), using 200 c.c. in all, until the alkaline liquid is no longer coloured red. The solution is then diluted to exactly 500 c.c., and 100 c.c. of this liquid further diluted to 1 litre. 350 c.c. of this solution, when diluted to 1 litre, should show a distinct cherry-red coloration against a white background, the tint being at least as deep as that of an alkaline solution of aloe-emodin of 1 part in a million (prepared by adding a trace of alkali to an aqueous solution of emodin of this strength). The above dilution corresponds to a yield of 2·8 per cent. of oxymethyl-anthraquinones from the rhubarb.

Good samples of the drug should yield from 2·8 to 4·0 per cent. The use of distilled water throughout the process is necessary, since the lime in tap-water precipitates the oxymethyl-anthraquinones. A. R. T.

Detection of Resinous Adulterants in Storax. Ahrens and Hett. (*Pharm. Centralb.*, xlv., 571; through *Pharm. Journ.*, 1904, lxxiii., 547.)—The authors base a test for the purity of storax on the ready solubility of resinous adulterants in cold petroleum-ether, genuine storax being only very slightly soluble. The storax is ground down in a mortar with sand and petroleum-ether, and the solvent evaporated. In the case of pure storax, a thick fluid residue is obtained, having an acid value of 40 to 55, and saponification value 180 to 197. In the presence of foreign resinous matter, the residue is resinous, has a terebinthinous odour, and shows an acid value of 116 to 121, and saponification value 172 to 178. A. R. T.

Constituents of Matico Oil. H. Thoms. (*Archiv. der Pharm.*, ccxlii., 328; through *Pharm. Journ.*, 1904, lxxiii., 428.)—A sample of Schimmel and Co.'s matico oil, of specific gravity 1·1343 at 16°, was found to contain a hydrocarbon solidifying at − 18° C., and boiling at 121° to 130° (13 m.m.); a small quantity of a phenolic ether, the bromo-derivative of which melts at 123° to 124° C.; and the two isomeric apiols, the so-called "dill apiol" being present to a much larger extent than the "parsley apiol." The "matico-ether" of Fromm and van Emster is stated by the author to be chiefly a mixture of the two isomeric apiols, while the maticoic acid of the same observers is a mixture of the two isomeric apiolic acids. Matico-camphor and asarone were absent. A. R. T.

Constituents of Essential Oil of Acorus Calamus. H. Thoms and R. Beckstroem. (*Berichte Pharm.*, xxxviii., 3187; through *Pharm. Journ.*, 1904, lxxiii., 428.)—The authors find the oil of sweet flag to contain free normal heptylic and palmitic acids; acetic and palmitic acids in the combined state; eugenol; asaryl aldehyde; asarone; and calameone ($C_{15}H_{26}O_2$), melting at 168° C., the crystalline body previously noted by Schimmel and Co., and by Soden and Rojahn. Asarone responds readily to the action of phosphoric and arsenic acids, the solid polymer par-asarone $(C_{12}H_{16}O_3)_3$ being produced with rise of temperature. Par-asarone forms a transparent vitreous mass at 173°, melts at 203°, and is readily soluble in most organic solvents. On evaporation, solutions of par-asarone leave a glassy residue having a crystalline structure. A. R. T.

Essential Oil of Ilex Paraguayensis. (*Haensel's Report*; through *Pharm. Journ.*, 1904, lxxiii., 453.)—The prepared twigs and leaves of this plant constitute the well-known Maté or Paraguay tea. The dried leaves of *Ilex Paraguayensis* yield 0·77 per cent. of a yellow oil, having in a marked degree the odour of the plant. The oil, which is solid at ordinary temperatures and melts at about 26·5° C., has the following analytical characters: Specific gravity, 0·8875; optical rotation for 100 millimetres, + 3·73° at 20° C.; acid value, 61; saponification value, 91. The oil has an acid reaction, and is readily soluble in 96 per cent., but only sparingly in 80 per cent., alcohol. A. R. T.

ORGANIC ANALYSIS.

Preservative for Starch Indicator. Frank X. Moerk. (*Amer. Druggist*, xlv., 144; through *Pharm. Journ.*, 1904, lxxiii., 453.)—The author has observed that if 2 c.c. of oil of cassia be dissolved, by shaking, in a litre of starch solution prepared in the usual manner the liquid is preserved indefinitely. This is a great advantage to those frequently using the indicator, and no objection has yet been found to the use of this preservative either in qualitative or quantitative work. A. R. T.

Invertin in Bees' Intestines. Axenfeld. (*Centralb. für Physiol*; through *Pharm. Journ.*, 1904, lxxiii., 428.)—Invertin is secreted in considerable quantities by the intestines of the bee, as well as of dipterous, coleopterous, and lepidopterous insects. Thus honey consists mainly of invert-sugar, even when the bees are fed with cane-sugar. A. R. T.

Volumetric Determination of Phenol. Frank X. Moerk. (*Amer. Druggist*, xlv., 144; through *Pharm. Journ.*, 1904, lxxiii, 453.)—The author has noticed that, in the determination of phenol by conversion into tri-bromophenol, and titration of the excess of bromine after addition of potassium iodide with thiosulphate and starch as indicator, the sharpness of the end-reaction is interfered with by the precipitated tri-bromophenol. Moreover, in the case of some old phenol solutions a bluish colour is acquired by the precipitate, which is not decolorized by excess of thiosulphate solution. These difficulties may be obviated by the addition of a small quantity of chloroform, added when the greater part of the brown iodine colour has been discharged by the thiosulphate. The precipitated tri-bromophenol is thus dissolved in the chloroform, and the end-point may be more easily observed. A. R. T.

Determination of Phenols in Essential Oils. (*Schimmel and Co.'s Report*, October-November, 1904, 129.)—The method described by S. B. Schryver (see ANALYST, 1900, xxv., 18), in which sodamide reacts with phenols with production of ammonia, is not applicable to the determination of the alcohols of essential oils. The results obtained by this method when applied to the alcohols are much too high, probably owing to the fact that the excess of sodamide produces more far-reaching changes in the alcohol molecule than in the case of phenols. Schryver found that the method could only be applied to those oils containing phenols, and free from ketones and aldehydes, and it is now apparent that the method is limited to those oils which are simply mixtures of phenols and terpenes. Thus the method gives good results in the cases of clove oil, pimenta oil, and artificial mixtures of phenols and terpenes. On the other hand, in oil of thyme, which contains the alcohols linalool and borneol in addition, thymol cannot be determined by this process. A. R. T.

Essential Oil of Nikkei. (*Schimmel and Co.'s Semi-Annual Report*, 1904, ii., 96.)—This oil is obtained from the leaves and young twigs of the Japanese cinnamon or cassia bark-tree (*Cinnamomum loureirii*), growing in the hottest provinces of Japan. The yield of oil is about 0·2 per cent., and the product has a bright yellow colour and a pleasant odour of citral and cinnamon. Nikkei oil has a specific gravity of 0·9005 at 15° C., an optical rotation of −8·75° for 100 millimetres, an acid value of 3·0, and an ester value of 18·6. It is soluble in 2 to 2·5 volumes of 70 per cent. alcohol with opalescence, and forms a clear solution with an equal volume of alcohol of 80 per cent. strength. The oil contains 27 per cent. of aldehydes (chiefly citral), about 40 per cent. of linalool, and some cineol. No cinnamic aldehyde was present. This constituent was found by Shimoyama in an oil from the root-bark of *Cinnamomum loureirii*, which oil had a greater density (0·982) than the present sample.

A. R. T.

West Indian Limette Oil. (*Schimmel and Co.'s Report*, October-November, 1904, 54.)—The composition of two authentic specimens of limette oil from Dominica is given in the following table:

	Hand-pressed.	Distilled.
Specific gravity	0·9008	0·8656
Optical rotation for 100 millimetres	+36·3°	+46·6°
Optical rotation of first 10 per cent. distilled	+39·5°	+53·1°
Acid number	6·05	1·80
Ester number	29·55	4·05
Residue on evaporation, per cent. ...	17·8	3·16

Both oils are soluble in 4 to 4·5 volumes and more of 90 per cent. alcohol, with slight cloudiness in consequence of the separation of a paraffin. The expressed oil shows in dilute alcoholic solution a faint blue fluorescence, probably due to the presence of methyl anthranilate. The oil has a golden-yellow colour, and a refreshing odour resembling that of lemon oil. The distilled oil possesses an unpleasant odour, resembling turpentine or pine-tar oil.

A. R. T.

Composition of Cypress Oil. (*Schimmel and Co.'s Report*, October-November, 1904, 22.)—This oil has been submitted to a further extended examination, and some hitherto obscure points cleared up. The following figures show the characters of cypress oil from two sources:

	Schimmel and Co.'s Distillate.	French Oil.
Specific gravity	0·8916	0·8680
Optical rotation for 100 millimetres ...	+16·5°	+26·5°
Acid number	1·88	—
Ester number	19·53	5·31
Ester number after acetylation ...	48·48	10·25
Solubility in 90 per cent. alcohol ...	1 : 2·5	1 : 5·5

In the high-boiling fractions of the oil the sesquiterpene lævo-cadinene is present. "Cypress-camphor" is an important constituent, and may be obtained from the oil by "freezing." Purified cypress-camphor crystallizes in long needles, which melt at 86° to 87° C. It is a sesquiterpene alcohol of the formula $C_{15}H_{26}O$, and distils at 290° to 292° C.

An intimate relationship between cypress-camphor and cedar-camphor (cedrol) has been generally assumed, and it has been found that the latter is the optically-active (dextro-rotatory) modification of the inactive cypress-camphor. By the action of formic acid cedar-camphor yields a lævo-rotatory, and cypress-camphor a dextro-rotatory hydrocarbon, $C_{15}H_{24}$. In other respects the two camphors are identical. A mixture of the two shows no depression of the melting-point, but melts uniformly at 86° to 87° C.

The following is a list of the known constituents of cypress oil: Furfural; dextro-pinene; dextro-camphene; dextro-sylvestrene; cymene; a ketone; sabinol (?); a terpene-alcohol (?); dextro-terpineol (melting-point 35° C.), probably as acetate; valeric acid; lævo-cadinene; a sesquiterpene-alcohol; cypress-camphor; and a body with a laudanum-like odour.

Cypress oil is having an extensive trial, with some success, as a curative agent in cases of whooping-cough. A. R. T.

Essential Oils of the Monardæ. (*Schimmel and Co.'s Semi-Annual Report*, 1904, ii., 57, 58, 97.)—J. W. Brandel (*Pharm. Review*, 1904, xxii., 153) found the dried herb of *Monarda citriodora* to yield 1 per cent. of a reddish oil having a specific gravity of 0·9437 at 20° C. The oil contained 65 per cent. of phenols, carvacrol and hydrothymoquinone being identified. About 1 per cent. of citral was present, and probably also some cymene.

The half-dried herb of *M. didyma* yields 0·04 per cent. of a golden-yellow oil of pleasant aromatic odour. It has a specific gravity of 0·8786 at 15° C., an optical rotation of $-24·6°$ for 100 millimetres, and is soluble in 1·5 to 2 volumes of 70 per cent. alcohol. This solution deposits a paraffin on further dilution. According to Brandel, the oil does not contain any appreciable amount of carvacrol or thymol.

The thymoquinone, which, together with hydrothymoquinone, is present in the oil from *M. fistulosa*, has been shown by F. Rabak (*Pharm. Review*, 1904, xxii., 190) to be produced by the action of an oxidizing ferment on the hydrothymoquinone. This soluble ferment may be isolated by bruising the fresh leaves in a mortar and stirring the product into a pulpy mass with water. The liquid on expression and filtration yields with alcohol a precipitate of the ferment. Rabak has shown that the oxydase reacts on the hydrothymoquinone with separation of dark-coloured crystals, which are gradually changed into thymoquinone. D. B. Swingle (*Pharm. Review*, 1904, xxii., 193) supposes the ferment to be identical with the β-katalase of Löw (*U.S. Dept. of Agric. Report.*, No. 68, 1). The ferment is destroyed at a temperature of about 74° to 78° C. A. R. T.

Composition of Rose Oils. Jeancard and Satie. (*Bull. Soc. Chim.* [3], xxxi., 934; through *Schimmel and Co.'s Report*, October-November, 1904, 79.)—The authors find the following analytical characters for an oil obtained from rose-blossoms after removal of the petals (*i.e.*, from the calyx, stamen, and pistil): Specific gravity at 15° C., 0·8704; optical rotation for 100 millimetres, $-41°$; solidifying point, $+8°$ C.; acid number, 6·12; ester number, 22·4. A stearoptene, consisting chiefly of a body melting at $+14°$ C., is present to the extent of 51·1 per cent. In addition, the oil contains also 13·6 per cent. of citronellol, which is the only alcohol present.

A. R. T.

Constants of Bulgarian Rose Oil. (*Schimmel and Co.'s Report*, October-November, 1904, 81.)—The following revised limits of composition for this oil are now stated: Specific gravity at 30° C. (water at 15° = 1·000), 0·849 to 0·862, rarely 0·863; optical rotation for 100 millimetres, − 1·5° to − 3·0°; refractive index at 25°, 1·452 to 1·464; congealing point, + 19° to + 23·5° C.; acid number, 0·5 to 3; ester number, 8 to 16; total alcohol (as geraniol), 66 to 74 per cent., exceptionally up to 76 per cent.; citronellol, 26 to 37 per cent., generally 30 to 33 per cent. The citronellol is determined by formylating the oil, the mixture, in the proportion of 1 volume of oil to 2 volumes of 100 per cent. formic acid, being heated for one hour over a reflux condenser. The method is then carried out as in the acetylation of oils.

A. R. T.

Some New Essential Oils. (*Haensel's Report*, July, 1904; through *Pharm. Journ.*, 1904, lxxiii., 548.)—Burdock root yields 0·18 per cent. of a brownish-yellow oil soluble in 80 per cent. alcohol, and having an acid reaction. The flowerless Conium herb gives about 0·08 per cent. of an acid, dark-brown oil of unpleasant odour. It deposits a stearoptene at low temperatures. Rectification with steam only yields 25 per cent. of distillate, the cooled residue being black and solidified. The brownish coloured rectified oil, which is soluble in 90 per cent. alcohol and acid in reaction, has nearly the same odour as the original oil, and contains free palmitic acid. Conium fruits yield only 0·018 per cent. of a blackish-brown oil of evil odour and neutral reaction. On rectification 51 per cent. distills as a greenish-yellow oil. The other characters of these oils are shown in the following table:

	Burdock Root.	Conium Herb.		Conium Fruit.	
		Crude.	Rectified.	Crude.	Rectified.
Specific gravity	0·9695 at 25°	0·9502 at 15°	0·9310 at 20°	0·8949	0·8313
Optical rotation (for 100 mm.)	+ 1·24° at 30°	—	—	—	− 2·16°
Acid value ...	13·5	60	—	—	—
Saponification value	236.8	70	—	34	—

A. R. T.

INORGANIC ANALYSIS.

On the Determination of Silver in Zinc and the Amount of Silver in Various Grades of Commercial Zinc. K. Friedrich. (*Zeits. angew. Chem.*, xvii., 1636.)—From 200 to 1,000 grammes of the granulated sample are treated with hydrochloric acid, preferably added in several portions, the zinc chloride obtained being poured off before the next lot of acid is added. As it has been found that, as soon as all the zinc has dissolved, the hydrochloric acid attacks the silver, the metallic residue is collected on a filter while some zinc is still present in it. It is then very carefully washed free from chloride, the presence of which would cause loss of silver in the subsequent operations, and, after ashing the filter, is run down with 7·5 to 15 grammes of assay lead and a little borax in a scorifier. The lead button obtained is cupelled, the silver weighed and examined as usual for gold. In order to allow for the loss of silver which always takes place during scorifying and cupelling, a quantity of pure silver, approximately equal to that contained in the sample taken, is run down in a scorifier with the same quantities of lead and borax as are taken for the main assay, and under as nearly as possible the same conditions, the lead button obtained being then cupelled and the loss in weight of the silver determined. This loss usually varies from 2·5 to 5 per cent. of the silver. In thirty-one samples of zinc examined, the quantity of silver present varied from 0·00011 to 0·00648 per cent. in the ordinary commercial grades, and from less than 0·000005 to 0·000014 per cent. in various kinds of "pure" zinc. Gold was present only in traces, if at all. The method used was shown to be reliable on a number of artificially-prepared zinc-silver alloys, some of which also contained the usual impurities of commercial zinc. A. G. L.

The Use of a Rotating Anode in the Electrolytic Estimation of Zinc. Leslie H. Ingham. (*Journ. Amer. Chem. Soc.*, xxvi., 1269.)—The author obtained good results in the electrolytic deposition of zinc from various solutions, using a platinum spiral revolving at the rate of 230 or 560 revolutions per minute as anode. The cathode consisted of a silvered dish, from which the zinc was removed at the end of each experiment by dilute sulphuric acid (1:50). It was found that, using an electrolyte containing sodium acetate, 0·5 gramme zinc would be completely deposited with the higher speed in fifteen minutes, using a current of 4 ampères (for about 100 square centimetres) and 11 volts. With sodium hydroxide solutions 0·25 gramme zinc could be deposited in fifteen minutes, using 5 ampères and 6 volts. From sodium formate solutions slightly acid with formic acid, 0·25 gramme zinc were deposited in twenty minutes, using 5 ampères and 8 volts. Potassium cyanide solutions gave unsatisfactory results.

Both the sodium acetate and formate methods were found to be especially applicable to the determination of zinc in zinc blende, the time required to complete the analysis on 0·5 gramme of ore being only about two and a half hours. The iron was removed as basic acetate or formate, the precipitate being dissolved in acetic or formic acid and reprecipitated, and the mixed filtrates were then electrolysed. The sodium hydroxide method could not well be used, but good results were obtained by precipitating the iron with ammonia in the presence of a large excess of ammonium chloride, and directly electrolysing the filtrate, using a current of 5 ampères and 5 volts. The anode was found not to lose in weight during these experiments. A. G. L.

The Electrolytic Deposition of Metals which Decompose Water from Solutions of their Salts. A. Siemens. (*Zeits. anorg. Chem.*, xc., 247.)—The author has made a number of experiments to see whether metals which decompose water could be obtained from aqueous solutions of their salts by electrolysis, by using a metal other than mercury to act as depolarizer at the cathode. He found that magnesium may be deposited together with nickel from solutions containing salts of both metals, provided the concentration of the magnesium is great. In dilute solutions, especially in the presence of ammonium salts, on the other hand, nickel may be quantitatively separated from magnesium. Magnesium may be similarly deposited with cobalt, less easily with iron, and only in traces with zinc. Aluminium and the metals of the alkaline earth group cannot be deposited at all in this way. The alkali metals may be deposited in small amounts together with nickel and tin, but not with iron or silver; ammonium appears to be deposited together with silver, but not with iron or nickel. In all these cases the metals in question are merely dissolved in the heavy metal, not combined with them.

Using high current pressures, the metals of the alkali and alkaline earth groups may be reduced from solutions of their anhydrous salts in acetone; magnesium, aluminium, and beryllium cannot be obtained in this way. A. G. L.

Electrolytic Separations Possible with a Rotating Anode. Donald S. Ashbrook. (*Journ. Amer. Chem. Soc.*, xxvi., 1283.)—The author has used an anode rotating at a speed of 300 to 400 revolutions per minute to effect the separation of a number of metals electrolytically. The dilution in each case was 125 c.c., and the cathode surface 100 square centimetres. It was found that in a sulphate solution containing 0·25 gramme of each metal and 1 c.c. of free sulphuric acid, *copper* could be very completely separated in ten minutes, using a current of 4 to 5 ampères and 1 to 4·8 volts, from iron, chromium, zinc, manganese, and uranium, but not from cobalt or nickel. In a nitrate solution containing 1 c.c. of free nitric acid, and using a current of 3 ampères and 4 to 5 volts, the same quantity of copper could be completely separated in twenty minutes from cadmium, arsenic, iron, chromium, uranium, manganese, and magnesium. From a solution containing 10 c.c. of phosphoric acid (specific gravity 1·085) and 50 c.c. of a 10 per cent. solution of Na_2HPO_4, using a current of 5 ampères and 6 volts, copper could be completely separated in ten minutes from iron, chromium, zinc, and magnesium, but not from nickel or cobalt; the deposit in each case contained a trace of phosphorus. It was not found possible to separate copper from antimony in tartrate solution. In a solution containing 25 c.c. of ammonia and 2·5 grammes of ammonium nitrate, using a current of 5 ampères and 7 volts, copper was completely separated in fifteen minutes from arsenic. With lead in nitric acid solution low results were always obtained for the copper.

Using similar solutions, it was found possible to separate completely, in sulphuric acid solution, *cadmium* from aluminium, iron, magnesium, manganese, and nickel, but not from chromium, which gave low, or from cobalt and zinc, which gave high, values; the current used was 5 ampères and 5 volts, the time being ten minutes. In phosphoric acid solution, using a current of 5 ampères and 7 volts, cadmium was completely separated in ten minutes from aluminium, chromium, iron, magnesium, and manganese, but not from cobalt, nickel, or zinc.

Silver can be quantitatively separated from aluminium, cadmium, chromium, cobalt, iron, lead, magnesium, manganese, nickel, and zinc, the current used being 3 ampères and 3·5 volts, and the time fifteen minutes. The deposits obtained, however, do not adhere at all well, and hence great care is required not to lose any silver in the washing. It was not found possible to use a potassium cyanide electrolyte.

Mercury was successfully separated in nitric and sulphuric acid solution from aluminium and magnesium, but not from other metals. The conditions are not stated in this case. A. G. L.

On the Preparation of Pure Sodium Hydroxide for Laboratories. F. W. Küster. (*Zeits. anorg. Chem.*, xc., 474.)—A large flat dish of porcelain or glass is filled with water to a depth of several centimetres, and a wide shallow bottle or other vessel of platinum, silver, or nickel placed in the centre. Above this, supported by a tripod, is placed a large funnel, closed at the bottom, made from pure nickel gauze. Into this funnel are put several hundred grammes of sodium at a time, and a bell-jar, slightly smaller in diameter than the flat dish, is immediately inverted over the whole, so as to shut off the sodium from the air by means of the water in the dish. To prevent the bell-jar from making too tight a joint, a few pieces of glass rod should be put on the bottom of the dish. As the water inside the jar evaporates, the sodium will gradually deliquesce, and the caustic soda formed drop into the bottle in the form of a 40 per cent. solution. The water in the dish is replaced from time to time; a little caustic soda may be added to it, more effectually to shut out carbon dioxide, but in practice this is not found necessary. The sodium hydroxide obtained in this way is free from the usual impurities of sodium, which are left behind in the gauze. It should be stored in large nickel bottles. As regards price, sodium hydroxide prepared in this way costs only about as much as the commercial "purified by alcohol" product. Nickel bottles may be obtained from Krupp of Berndorf. A. G. L.

Preparation of an Alcoholic Solution of Potassium Hydroxide which will remain Colourless. H. Thiele and R. Marc. (*Zeit. öffentl. Chem.*, 1904, x., 386, 387.)—The authors recommend the use of potassium sulphate and barium hydroxide for the preparation of this solution. To make 1 litre of $\frac{N}{2}$ solution, 43·5 grammes of pure potassium sulphate and 110—120 grammes of barium hydroxide are triturated in a platinum or china basin with 100 c.c. of water. The basin and its contents are weighed and then heated to boiling for fifteen minutes, with constant stirring. After cooling, water is added until the original weight is again obtained. The contents of the basin are now rinsed into a large flask with 800 c.c. of alcohol,* and another 100 c.c. of water added. From 3 to 4 c.c. of a saturated solution of potassium sulphate are now introduced; the mixture is well shaken and allowed to settle. A small quantity of the clear supernatant solution is tested with sulphuric acid to ascertain its freedom from barium hydroxide, and the whole is then drawn off. The solution will be of approximately the desired strength, and will remain clear and colourless for months. W. P. S.

Note on a Reaction of Potassium Ferrocyanide. A. Gutbier. (*Zeit. anorg. Chem.*, xli., 61.)—If to an aqueous solution of potassium ferrocyanide an aqueous solution of phenylhydrazine hydrochloride is added no reaction takes place in the cold. On warming, however, a light brownish-red colour is produced, which on increasing the temperature, changes through a light-red to a dark-red colour, suddenly disappearing at the same time that a yellowish-green precipitate is produced, and nitrogen and hydrocyanic acid are evolved. By means of steam distillation a dark oil of unknown composition may be separated from the liquid. A. G. L.

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* From the context it appears that ordinary commercial alcohol is intended.

The Determination of Iodide in the Presence of Bromide and Chloride. E. Thilo. (*Chem. Zeit.*, 1904, xxvii., 866.)—The author finds that when silver nitrate solution is added to a mixture of chlorides, bromides, and iodides, precipitation occurs in three stages. Silver iodide is first formed. When this is completely separated, silver bromide comes down, and finally the chloride. The exact point at which the iodide is completely precipitated is fixed by placing a drop of the solution on a strip of filter-paper which has been immediately before dipped in palladium chloride solution. In the presence of the smallest trace of iodide a dark brown stain is formed, and the end-point is sharp to at least 0·1 c.c. of $\frac{N}{10}$ silver solution.

Bromide is determined in a precisely similar manner, continuing the addition of silver until no stain is obtained by spotting on gold chloride paper; or chromate can be added and silver solution run in until the colour change indicates complete precipitation of the bromide and chloride, when the silver chromate is dissolved by the addition of a little nitric acid, and the mixed chloride and bromide filtered in a Gooch crucible and weighed. The excess of silver in the filtrate is determined by titration with sulphocyanide, after reduction of the chromate by ferrous sulphate. From the weight of the mixed precipitate, and the amount of silver present, the proportions of silver chloride and bromide may be calculated. H. A. T.

A Method for the Determination of Chloric Acid. W. S. Hendrixson. (*Amer. Chem. Journ.*, xxxii., 242.)—The chloric acid is reduced to hydrogen chloride by means of metallic iron in fairly strong sulphuric acid solution at room temperature. The reaction requires about one hour. When reduction is complete, the chloride formed is determined by Volhard's method, nitric acid being added to oxidize the ferrous salt. The method gives good results, and is applicable to bromates as well as chlorates. Any perchloric acid present remains unreduced, and consequently does not interfere. A. G. L.

The Electrolytic Determination of Nitric Acid with a Rotating Anode. Leslie Howard Ingham. (*Journ. Amer. Chem. Soc.*, xxvi., 1251.)—The author reduces the nitric acid to ammonia by electrolysing the solution in presence of copper sulphate; a known amount of sulphuric acid being added, and the copper sulphate containing a known amount of acid, the quantity of ammonia formed is determined by titration. The conditions found to give the best results are as follows: 0·5 gramme of potassium nitrate is dissolved in water, 25 c.c. of standard sulphuric acid and 25 c.c. of copper sulphate solution, containing 0·2533 gramme of copper, are added; the total volume is made up to 125 c.c., and a current of 4 ampères is passed through for thirty minutes, the voltage at the outset being 10 and at the end 17 volts. Apparently the speed of the rotating anode is 230 revolutions per minute. At the end of the operation the liquid is titrated with standard ammonia, using litmus or methyl orange as indicator. In ten determinations made in this way the percentage of nitrogen found varied from 13·79 to 13·94, with a mean of 13·865, the theoretical value being 13·86 per cent. A. G. L.

Analysis of Liquid Carbon Dioxide. O. Wentzki. (*Zeit. öffentl. Chem.*, 1904, x., 385, 386.)—It has recently been shown by Woy (Analyst, this vol., p. 323) that the first portion of carbon dioxide drawn from an upright full cylinder of the liquid contains less air than the immediately succeeding portions, and that the quantity of air then gradually diminishes until pure carbon dioxide is obtained. The author considers that these results are simply due to the temperature of the contents of the cylinder. At temperatures below 22° C. a certain amount of gaseous carbon dioxide is always present in the cylinder, and this contains considerably more air than the liquid portion. At 25° C., however, an ordinary full cylinder is completely filled with liquid carbon dioxide, and a portion, on being withdrawn, gives the average percentage of air in the whole. The withdrawal diminishes the temperature and the pressure, and some of the liquid is consequently converted into gas, in which the air present concentrates. A second sample taken from the cylinder will therefore contain more air than the first sample. W. P. S.

The Determination of the Oxygen Absorption of Waters containing Large Amounts of Chlorides. Ruppin. (*Zeit. Untersuch. Nahr. und Genussmittel*, 1904, viii., 418.)—In the determination of the reducing power of waters towards permanganate, untrustworthy results are obtained in the case of highly saline waters, such as sea-water, even when alkaline permanganate solution is employed (see Analyst, 1902, 341), owing to the liberation of chlorine when the solution is acidified at the end of the operation. The author describes a device for preventing this loss, consisting in a hollow stopper ground into the neck of the flask in which the reaction is carried out; a bent tube, provided with two bulbs, leaves the top of this stopper, and the bulbs are partially filled with a 10 per cent. solution of potassium iodide, to form a trap for any chlorine liberated when the solution in the flask is acidified. After acidification, and when the colour of the permanganate has disappeared, potassium iodide solution is added through the trap-tube, and, after cooling, the mixed contents of the bulbs and flask are titrated with thiosulphate solution as usual.

W. P. S.

Quantitative Determination of Phosphorus in Solutions. A. C. Christomanos. (*Zeits. anorg. Chem.*, xc., 305.)—To determine phosphorus dissolved in ether or benzene, a portion of the liquid is weighed out into a tared flask, and an excess of a 10 per cent. solution of copper nitrate in water is added. The whole is well shaken for a few minutes, and then heated on a water-bath for fifteen or twenty minutes to drive off the ether or benzene. With continuous shaking and heating of the flask bromine is next added drop by drop until the whole of the copper has been converted to cupric bromide, phosphorous and phosphoric acids being formed at the same time. The liquid is then evaporated to a low bulk, after the addition of nitric acid, to expel the bromine and oxidize the phosphorous acid, an excess of ammonia is added, and the determination completed as usual. A. G. L.

A Radically New Method for the Determination of Sulphur in Iron and Steels. H. B. Pulsifer. (*Iron and Steel Magazine; through Chem. News*, xc., 230.)—The method essentially consists in bringing the steel into solution by a mixture of chloric, hydrofluoric, and hydrochloric acids, the insoluble residue being fused with sodium peroxide and the solution of the melt added to the main solution, in which the sulphuric acid is then precipitated as usual. In a 250 c.c. broad Jena beaker 2·5 grammes of the sample are moistened with water and then treated with 20 c.c. chloric acid (specific gravity 1·12) and a very little hydrofluoric acid. As soon as the first very violent reaction is over, 5 c.c. hydrochloric acid are added, the beaker is covered, and the whole heated to boiling to complete solution. The insoluble residue left is next filtered off, washed two or three times with a little water, dried by suction, and placed in a 20 c.c. nickel crucible. Sodium peroxide is added to it, and, whilst the cover is held down with the tongs, the crucible is heated by a strong flame. The action is over in a few seconds, and as soon as the melt is solid it is dissolved in 50 c.c. water and a little hydrochloric acid, any unburnt carbon being filtered off. The filtrate is added to the main filtrate, which in the meantime has been boiled down, after the addition of 20 c.c. concentrated hydrochloric acid, to a volume of less than 10 c.c. The total volume of the mixed solutions should be about 100 c.c.; barium chloride is added, and the determination finished as usual.

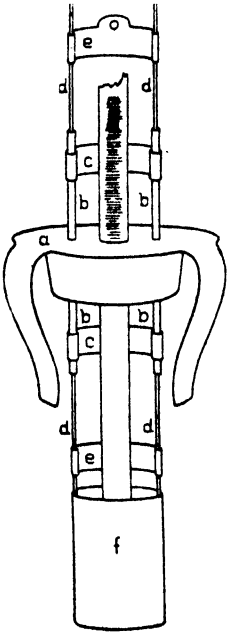
Values found by this method for sulphur in steels generally appear to be considerably higher than those given by the older methods; for cast-irons the agreement seems to be good. The proportion of sulphur in the insoluble residue, which the author treated separately for the test analyses, is considerable, in one case being over 40 per cent. of the total sulphur. A. G. L.

On the Use of Phosphorous Acid for the Determination of Selenium and Tellurium. A. Gutbier. (*Zeits. anorg. Chem.*, xli., 448.)—From the analyses quoted, it appears that phosphorous acid can be successfully used for the determination of selenium and tellurium when these elements are in the tetravalent condition; solutions of selenic and telluric acid, on the other hand, are not reduced appreciably by phosphorous acid. The method used is to add a concentrated solution of phosphorous acid to a solution of selenium or tellurium dioxide in 20 per cent. hydrochloric acid and to boil for a short time; the precipitate is filtered on a Neubauer platinum crucible, dried at 105° C., and weighed. A. G. L.

A Comparative Examination of the Gravimetric Methods for the Determination of Selenium. A Gutbier, G. Metzner, and J. Lohmann. (*Zeits. anorg. Chem.*, xc., 291.)—The authors have made a comparative study of the various methods of reducing selenium from solutions of the dioxide. In all cases they have found it necessary to boil the solution with an excess of the reducing agent, and then, after apparently the whole of the selenium had been precipitated, to add a little more of the reagent, and to boil for another ten minutes. The selenium obtained was always dried at 105° C. and weighed. It was found that hydrazine hydrate gives very good results, but a very concentrated solution of selenium dioxide must be employed. This is less necessary in the case of hydrazine hydrochloride or sulphate, which also give good results; with the last reagent it is necessary to acidify the liquid with hydrochloric acid. Sulphurous acid in hydrochloric acid solution also gives very good results, but large quantities of these reagents must be used. Alkali sulphites are not to be recommended, as selenium obstinately retains salts. Hydroxylamine hydrochloride gives good results only under certain conditions, and very prolonged boiling is required to complete the reaction. Potassium iodide in hydrochloric acid necessitates the use of dilute solutions, and high results are invariably obtained owing to the retention of potassium chloride by the selenium. Finally, the use of hypophosphorous acid in alkaline solution suffers from the same defects, and, further, precipitation is never quite complete. A. G. L.

APPARATUS.

A Modification of the Freezing-point Method. S. W. Young and W. H. Sloan. (*Journ. Amer. Chem. Soc.*, xxvi., 913.)—Instead of following the usual custom of slightly supercooling a liquid, allowing ice to form, and taking the highest temperature reached during this stage as the melting-point, the authors prefer to stir a liquid slightly above its solidifying-point with ice, and to consider the lowest temperature reached as the melting-point, since the ice will continue to melt and cool the liquid until equilibrium between the two is established. To carry out the method, a vacuum-jacketed tube, 260 millimetres long and 45 millimetres in internal diameter, is used. On the top of this rests a brass plate, *a* (see figure), provided with three prongs, which grip the vacuum-tube firmly. On the under-side of this plate is cemented a cork which fits the mouth of the tube. Both plate and stopper are perforated so as to carry a Beckmann thermometer. At either side of this a brass tube, $\frac{1}{8}$ inch in diameter, soldered on to the plate, passes through the plate and stopper (*b*, *b*). These tubes serve as guides for the upright rods of the stirrer frame (*d*, *d*). Brass strips (*c*, *c*, *e*, *e*) serve as braces to keep the arrangement rigid. At the upper brace (*e*) connection is made with a rod connected with a wheel which moves the stirrer. At the lower end of the rods is soldered the cylindrical roll of silver-foil on which the ice is to be frozen (*f*).



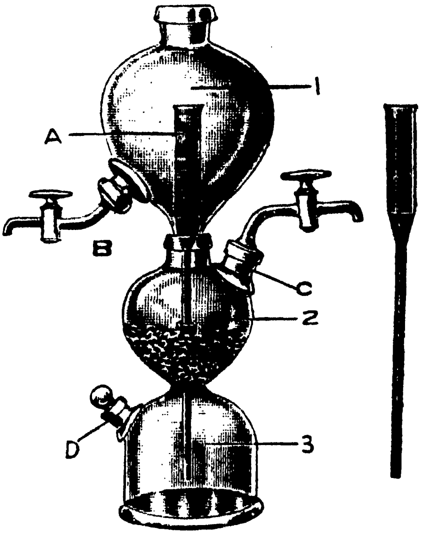
To freeze the ice on the stirrer, the latter is placed in a glass tube slightly smaller than the vacuum-tube. This tube is fitted with a stopper carrying another piece of glass tubing, slightly larger than the thermometer. Cooled boiled water is poured into the outer tube, and frozen by immersing the whole in a freezing-mixture. The stirrer with the ice is then easily removed by warming the two glass tubes slightly. After removal it is placed in a second vacuum-jacketed tube, to which it is also transferred between any two determinations. One stirrer can be used six to eight times.

To make a determination, about 125 c.c. of water, cooled to nearly 0° C., are placed in the vacuum-jacketed tube, which is then put into a vessel containing ice.

The thermometer and stirrer are then inserted, and the stirrer is set in motion unti a constant reading is obtained. Stirrer and thermometer are then removed, a weighed amount of substance is introduced, the stirrer and thermometer replaced, and another reading taken. The whole apparatus is then taken out of the outer vessel, the stirrer and thermometer are removed after allowing them to drip for a moment, and the vacuum-tube and solution weighed. By subtracting the weights of the empty tube and of the substance added, the weight of water present is obtained, from which the concentration is calculated. This procedure is repeated several times.

The results obtained in this way for the molecular lowering are very concordant, slightly too low, and decreasing with increasing concentration of the solution, owing to the increasing amount of radiation to the outer vessel. The authors discuss some of the sources of error, and hope to eliminate these so as to obtain still better results. A. G. L.

An Improved Form of Kipp's Apparatus. H. J. Friswell. (*Chem. News*, xc., 154.)—The apparatus shown in the figure differs from the usual form of Kipp in having a neck and cock (B) on the upper bulb (1), and also in being fitted with the tube A, which reaches from about three-quarters of the height of 1 nearly to the bottom of the lowest bulb. This tube is fixed to the bulb 1 by means of a ground-glass joint, warmed and smeared with marine glue before use. The bulb itself is fitted into the middle bulb, as usual, by a glass joint. To charge the apparatus, zinc, etc., in fairly large pieces is placed in bulb 2; a strong solution of zinc sulphate, etc., according to the gas to be generated, is then poured into the lowest bulb up to within ½ inch from the top; and acid is then poured into the top bulb so as to stand an inch or so above the top of tube A. As soon as the stopcock at C is opened, the acid will flow down A into the lowest bulb, and, being lighter than the heavy salt solution therein, will rise through it to the middle bulb. When the stopcock is shut the acid is forced downwards, and causes the salt solution to overflow from A into the upper bulb, where it falls through the lighter acid. As the acid becomes exhausted, the salt solution will tend to approach the top of tube A in the upper bulb, and may be drawn off through B, fresh acid being filled in through a funnel long enough to enter A for a short distance. To recharge the material in bulb 2, enough salt solution is drawn off through B to uncover the top of A, from which the greater part of the liquid is next removed by means of a long-stemmed pipette, after which the zinc, etc., is put in as usual through C; the neck D need never be used at all.



The apparatus is said to be less wasteful and easier to recharge than the ordinary Kipp form. As its economical working depends on the difference in specific gravity of two liquids in contact with each other, it is evident that it must not be moved about more than is absolutely necessary; the falling of small fragments of the solid material into the lowest bulb should also carefully be avoided. A. G. L.

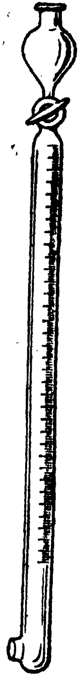


Fig. 1

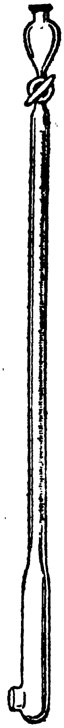


Fig. 2

Improved Measuring-Tube for Dumas' Nitrogen Process. Anton Landsiedl. (*Chem. Zeit.*, 1904, xxviii., 643.)—Greater simplicity and ease of working are claimed for this form of receiver. The measuring-tube is filled with mercury through the short lateral tube in the usual way, and the soda solution run in from the cup or funnel. Any air can be expelled by closing the orifice with the thumb, and admitting a little mercury from the cup, displacing the air.

An additional advantage lies in the fact that, should this be necessary, further soda can be added during the operation without danger of loss; also that, after emptying out the mercury and soda, the tube can be washed out with distilled water before being brought into the final measuring-cylinder.

The apparatus may be obtained from Dr. H. Goekel, Berlin.

H. A. T.

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REVIEWS.

Percentage Tables for Elementary Analysis. By Leo F. Guttmann, Ph.D., A.I.C. London: Whittaker and Co.

By the aid of these tables the percentages of carbon and hydrogen in the substance under examination are readily calculated from the amounts of carbon dioxide and water obtained in a combustion analysis, and hence the tedious operation of working out several sums in proportion is avoided. As the tables substitute addition and subtraction for multiplication and division, accurate results are more easily attained than by the older method. The tedious task of compiling the tables has been well done; they have been carefully checked, the proofs scrupulously revised, and they are of guaranteed accuracy. The tables cannot fail to be most useful to those engaged in organic analysis, and form a welcome addition to the list of those commendable labour-saving books which have appeared of late years. A table for the reduction of volumes of nitrogen to grammes is appended. W. J. S.

Inks: their Composition and Manufacture. By C. Ainsworth Mitchell, B.A. (Oxon.), F.I.C., and T. C. Hepworth. Pp. xiv and 251, with 46 Illustrations, including 4 Plates. London: Charles Griffin and Co., Ltd., 1904. Price 7s. 6d. net.

To all who are interested in the history and manufacture of writing and printing inks this is a most useful and interesting volume. Its scope differs from the work on inks issued in 1902 by S. Lehner, and includes three sections: Writing Inks;

Printing Inks; and Inks for Miscellaneous Purposes, including copying, safety, marking, and other inks for special purposes. Curiously, there is no reference in the book to typewriting inks.

The history of ancient writing and inks is very good, with illustrations of early Egyptian writing on papyrus. Indeed, throughout the book it is exceedingly well illustrated, showing the various tannin and other ink materials more fully, I believe, than is done in any other work of the same compass. The tests and processes given for the examination of materials imply a considerable knowledge of chemical laboratory methods, and will be most useful to those who have had the requisite training.

Under Printing Inks, the most recent methods employed in their manufacture and the machinery are fully described and figured, including the process of three-colour printing by which wonderful effects are obtained. The colour diagram shown here is of great interest.

The book is well got up, and very few slips occur in the printing. The register of English patents relating to inks appears a very full and complete one, and the careful way in which information has been collected from widely-scattered sources renders the book a valuable one to all who take up this subject. T. F.

APPLICATION OF SOME GENERAL REACTIONS TO INVESTIGATIONS IN ORGANIC CHEMISTRY. By DR. LASSAR-COHN. Translated by Dr. J. BISHOP TINGLE. 101 pp. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. Price 4s. 6d. net.

The subject-matter of this little work, if divorced from deplorable blemishes of translation, is excellent. It aims at assisting the investigator to gauge, before application, the comparative value of the numerous processes which may be used for effecting any particular change necessary to the progress of his research. This, however, is not a requirement of students until they become investigators, and the present volume would quickly demoralize an individual who has not acquired a respectable literary style. Split infinitives may leave veterans unscathed, but they are bad for beginners. When the translator writes "tetrabromoparaxylene can only be converted into tetrabromoterephthalic acid, to a very limited extent, by any of the ordinary oxidizing agents," and then proceeds to mention an extraordinary oxidizing agent which effects the same change, he is at least intelligible, if ungrammatical; but what is the exact significance of "tertiary isobutylglyceryl-β- is hydroxylamine obtained from nitroisobutylglycerol" (p. 56)? "Hyacyl," whatever it means, may be an accident, but "hydrhaloïd" seems intentional, and the intention is too Carrolian for use in text-books. "Phenylsemicarbazine" is not English, though its meaning is clear, and the same remark applies to the employment of "substitution" when the idea of "replacement" is intended. "Rescorcinol" is probably a misprint, but "brom acid" is slang, and "phenyl glycocoll" in the line next to "phenylaminoxalic acid" is carelessness. The work of Aschan is familiar to most readers of chemical literature, but who is "Ashan," a gentleman mentioned twice in the text and also in the index? In these days of universal charity the translator's hope "that, in course of time, the blind, trial and failure process may be increasingly restricted" (p. 2) will be condemned as heartless, and must not be taken too seriously. In every five pages something of this order will be found to exasperate the reader, and distract his mind from the really valuable subject-matter; but enough has been said to indicate that no conscientious teacher should place the book in the hands of a student without grave warnings against its lamentable want of style. M. O. F.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

Pass List of the January Examinations.

Of eighteen candidates who entered for the Intermediate Examination, the following thirteen passed: S. J. M. Auld, Ph.D. (Würzburg), S. L. Archbutt, A. W. Bain, B.A., B.Sc. (London), J. T. Cart, B.Sc. (London), T. F. Cowie, D. Gair, B.Sc. (London), C. T. Gimingham, H. G. Harrison, B.A. (Cantab.), Elsie S. Hooper, B.Sc. (London), S. G. Liversedge, W. H. Simmons, B.Sc. (London), A. S. Stockwin, B.Sc. (London), and E. J. Wilson, B.A. (Cantab.). In the Final Examination for the Associateship (A.I.C.) in Mineral Chemistry, of six who entered, the following five passed: H. Calam, B.Sc. (Victoria), J. D. Fraser, R. F. Korte, Ph.D. (Heidelberg), W. D. Rogers, Assoc.R.C.Sc. (London), and P. E. Spielmann, Assoc.R.C.Sc. (London). Of three Candidates in Organic Chemistry, one passed: H. E. Laws, B.Sc. (London); and of nine who entered in the Branch of the Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following six passed: G. S. A. Caines, S. W. Collins, R. M. Filmer, H. E. Gresham, B.Sc. (London), V. H. Kirkham, B.Sc. (London), and J. Miller. One Candidate passed a General Practical Examination for the Fellowship (F.I.C.): W. H. Merrett, A.R.S.M.

The Examiners in Chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. Dr. A. P. Luff conducted the Examination in Therapeutics, Pharmacology, and Microscopy.

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MARCH, 1905. Vol. XXX., No. 348.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Monthly Meeting of the Society was held on Wednesday evening, February 1, in the Chemical Society's Rooms, Burlington House. In the absence of the President, Mr. Bertram Blount occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Edward Hinks, B.Sc. (Lond.), F.I.C., 23, Tavistock Road, Croydon, assistant to Sir Thomas Stevenson; and Samuel Ernest Melling, F.I.C., 359, The Cliff, Higher Broughton, Manchester, analytical and consulting chemist, were read for the first time.

Messrs. W. Ackroyd and J. H. Johnston were elected members of the Society.

The following papers were read: "The Volumetric Estimation of Reducing Sugars," by Arthur R. Ling and T. Rendle; "The Inversion of Cane-sugar in the presence of Milk Constituents," by the Hon. Francis Watts, D.Sc., and H. A. Tempany, B.Sc.; and "The Colorimetric Estimation of Salicylic Acid in Food-stuffs," by F. T. Harry and W. R. Mummery.

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NOTE ON COMMERCIAL AMYL ALCOHOL.

BY H. DROOP RICHMOND, F.I.C., AND J. A. GOODSON.

(Read at the Meeting, December 7, 1904.)

A CONSIGNMENT of amyl alcohol was found to give, when used in the Gerber method of fat estimation, constantly high results. With milks the average reading was 0·10 per cent. higher than gravimetric estimations, and with separated milks (containing about 0·1 per cent. of fat) 0·06 per cent. higher. This amyl alcohol complied with the specification laid down by one of us in conjunction with F. R. O'Shaughnessy, and when 1 c.c. was treated with 11 c.c. of water and 10 c.c. of sulphuric acid, only the faintest indication of an insoluble layer was seen on the surface.

Various mixtures of amyl alcohol acid and water were made, and the layer read off in a Gerber bottle as if it were fat.

1	c.c. amyl alcohol,	11	c.c. of water, and	10	c.c. of acid yielded	trace.
2	,, ,,	10	,, ,,	10	,, ,,	0·12
3	,, ,,	9·5	,, ,,	9·5	,, ,,	0·04
4	,, ,,	9	,, ,,	9	,, ,,	trace.
5	,, ,,	8·5	,, ,,	8·5	,, ,,	none.

The impurity in this alcohol was soluble in the acid mixture, and the solubility increased with the amount of amyl alcohol.

250 c.c. were distilled through a fractionating column:

94° to 124·5° (corrected).	16 c.c distilled.
124·5° to 127·5° ,,	32
127·5° to 128·5°	36
128·5° to 129·5°	62·5
129·5° to 130·5°	73
130·5° to 131·5°	18·5
Leaving a residue of	10 c.c.

When 2 c.c. of each fraction were treated with 10 c.c. of water and 10 c.c. of sulphuric acid, no layer was observed to rise; but 2 c.c. of the residue gave a layer measuring 3·0, which had a smell of petroleum. The residue from the distillation of 500 c.c. was shaken with sulphuric acid, the layer separated, washed with water, caustic soda solution, and again with water, and dried with calcium chloride. This had a density of 0·803, and boiled at 190° to 230°, smelt of petroleum, and was slightly fluorescent.

Mr. J. B. P. Harrison informs us that petroleum barrels are used as packages for amyl alcohol, and that as much as 4 per cent. of petroleum has been observed.

A milk which read 4·32 per cent. fat with the crude alcohol gave an average of 4·21 per cent. with various fractions of the distilled product, and a separated milk 0·17 per cent. with the crude and 0·12 per cent. with the distilled.

Mixtures of amyl alcohol with varying amounts of petroleum were made:

Percentage of Petroleum.	Reading with 2 c.c. Alcohol, 10 c.c. Water, 10 c.c. Sulphuric Acid.	Milk.	Separated Milk.
4·8	0·67	4·48	0·48
2·4	0·25	4·37	0·25
1·2	0·11	4·23	0·16
0·6	trace	4·19	0·12
0·3	doubtful trace	4·12	0·09
none	none	4·10	0·09

We also found that the drying of the fat obtained by the method described by one of us with C. H. Rosier (ANALYST, 1899, xxiv., 172) took some hours before a constant weight could be obtained when this amyl alcohol was used.

We conclude that:

1. The amyl alcohol in question contained about 1 per cent. of petroleum.
2. This quantity of petroleum is soluble in a mixture of the amount of amyl alcohol, sulphuric acid and water used in the Gerber method, and is partially extracted by small amounts of fat (0·1 per cent.), and wholly by larger amounts.
3. Amyl alcohol which gives any visible insoluble layer when 2 c.c. are treated with 10 c.c. of water and 10 c.c. of sulphuric acid should not be used in the Gerber method; a blank Gerber test is insufficient.

Van Haarst (ANALYST, 1903, 213) and Siegfeld (ANALYST, 1904, 113) have stated that apparently pure amyl alcohol may give high results with Gerber's method, and it is probable that they were dealing with amyl alcohol containing small amounts of petroleum.

Tested according to specification :

Specific gravity at 15·5° ... ... 0·8149
Distillation of 25 c.c. ... ... first drop at 125° C.
125 to 127·5 2·25 c.c.
127·5 to 130·5 21·25 c.c.
Final temperature ... ... 131°
10 c.c. and 10 c.c. HCl ... clear colourless solution.
0·5 c.c. water produces turbidity.
Polarization ... 2·58° in 200-millimetre tube $[\alpha]_D = 1{\cdot}61°$.
Blank Gerber ... faint trace of layer.

Discussion.

Mr. Richmond, in reply to a question put by the President, said that in the sample referred to the proportion of petroleum was, they had concluded, somewhere about 1 per cent. This conclusion was based, not only on the figures given, but also on the amount they had been able to separate by fractional distillation. He had, however, heard of a sample containing 4 per cent. of petroleum.

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THE DETECTION AND ESTIMATION OF SMALL QUANTITIES OF MALTOSE IN THE PRESENCE OF DEXTROSE.

By JULIAN L. BAKER, F.I.C., and W. D. DICK, F.I.C.

(*Read at the Meeting, November* 2, 1904.)

Last year the Council of this Society honoured us by suggesting that we should submit L. Grimbert's method for detecting small quantities of maltose in presence of dextrose (*Journ. Pharm. Chim.*, 1903, xvii., 225, and this Journal, 1903, 151) to a critical examination.

Lepine and Boulud (*Comptes Rend. Soc. Biol.*, December, 1901) devised a method for detecting dextrose in presence of maltose which consisted in treating the phenyl osazones of these sugars with ether. It was claimed that maltosazone was dissolved and could be recognised by its crystalline form. Grimbert (*loc. cit.*), however, stated that maltosazone is as insoluble in ether as dextrosazone. He recommended the following method as capable of detecting maltose in a 0·1 per cent. solution containing 0·1 to 1 per cent. of dextrose: 20 c.c. of the solution containing the two sugars are treated with 1 c.c. of phenylhydrazine and 1 c.c. of glacial acetic acid, and the whole heated on the water-bath for an hour, and then cooled. The mixed osazones are washed with cold water and dried, then washed with benzene until the washings are colourless, and dried at 100° C. They are then triturated in a mortar with the smallest possible amount of acetone diluted with its own volume of water, and, on allowing the filtrate to stand, crystals, with the characteristic form and melting-point (196° to 198° C.), are deposited. Or, the osazones may be heated in a minute amount of water for five minutes on the water-bath, and then rapidly filtered, leaving the dextrosazone, which is much more insoluble in hot water, on the filter.

In the course of this investigation we employed freshly distilled phenylhydrazine and pure maltose and dextrose. To test Grimbert's method systematically, pure specimens of maltosazone (melting-point, 195° to 200°C.) and dextrosazone (melting-point, 200° to 202° C.) were prepared and the relative solubilities examined in different solvents.

Solvent.	Maltosazone.	Dextrosazone.
Boiling water	Completely soluble	Slightly soluble.
Ether (cold) ...	Slightly	
Benzene		
Toluene		
Xylene		
Chloroform		
Acetone ,, ...	Very	Fairly
50 per cent. aqueous acetone (cold)	,,	Slightly
20 per cent. aqueous acetone ,,	Fairly	Insoluble.
10 per cent. aqueous acetone ,,	Slightly	Insoluble.

It will be noticed that 50 per cent. aqueous acetone dissolves dextrosazone to some extent, and, therefore, throughout these experiments we used 20 per cent. acetone.

The solubility of pure maltosazone in benzene, in our opinion, precludes the use of this solvent for removing the tarry matters produced during the formation of the osazones, for it was found that when a prepared mixture of the pure osazones of maltose and dextrose, in which the former was present in small quantity, was washed with cold benzene the maltosazone was for the most part dissolved. Moreover, the solubility of the impure osazones of maltose and dextrose, such as results when the mixed sugars are converted into osazones, is very marked in cold benzene. This portion of Grimbert's process had to be abandoned, and we endeavoured to prepare the osazones in such a manner that the tarry products of the reaction were reduced to a minimum. Clean osazones may be obtained by heating 20 c.c. of 5 per cent. solution of the mixed sugars contained in a round-bottomed flask fitted with a tube condenser with 1 gramme of freshly distilled phenylhydrazine and 1·5 c.c. of 50 per cent. acetic acid for three-quarters of an hour in a boiling water-bath. The osazones are then cooled and filtered through a Gooch crucible, washed with 25 c.c. of cold water, and then with 10 c.c. of cold 5 per cent. aqueous solution of acetone. When the preparation is well drained, it is mixed in the Gooch crucible with 10 to 15 c.c. of cold 20 per cent. acetone, and filtered. On standing, the filtrate deposited a mixture of maltosazone and a little dextrosazone. The former, if present in sufficient quantity, could be easily identified, when examined under the microscope with a ¼-inch or ⅛-inch objective, by its characteristic ribbon-like crystals. Working under these conditions, dextrose, when mixed with 15 per cent. of maltose, gave definite indications of maltosazone, 10 per cent. of maltose was doubtful, and no satisfactory separation of maltosazone could be obtained when dextrose was mixed with 2½ and 5 per cent. of maltose.

When mixtures of the pure osazones of maltose and dextrose were made in varying proportions and triturated with 20 per cent. acetone, it was easy to detect 5 and 10 per cent. maltosazone in the filtrate. This illustrates the influence which

the by-products of the osazone reaction has on the crystalline structure of maltosazone, and this would be still more marked if the sugars themselves were impure. The difficulty of preparing pure maltosazone from the products of the hydrolysis of starch is well known.

Attempts were made to estimate the amount of maltose in a mixture of this sugar and dextrose by converting into the osazones in the manner described above. After washing with the 25 c.c. of cold water the osazones were dried until constant in weight in the water-oven, and then washed with 20 c.c. of cold 20 per cent. acetone. The osazone was again dried and weighed, and it was hoped that when working under these standard conditions the difference in weight due to the loss of maltosazone might be a measure of the maltose present. The results were very variable, and, after many trials, we were forced to the conclusion that no process of estimation could be based on the difference in solubility in 20 per cent. acetone of maltosazone and dextrosazone.

We next tried to see if it was possible to estimate small quantities of maltose by acid hydrolysis. Numerous experiments were carried out to determine the proper conditions for the inversion of maltose. When 10 c.c. of a 2 per cent. solution of maltose are heated in a flask fitted with a tube condenser with 1 c.c. of strong hydrochloric acid (specific gravity 1·16) and 10 c.c. of water for ninety minutes in a boiling water-bath, the contents of the flask being then cooled and neutralized, the inversion was completed to the extent of 96·5 per cent. As the object of the investigation was to estimate *small* quantities (from 2½ to 15 per cent.) of maltose in presence of dextrose, this amount of uninverted maltose would be without any marked influence on the results. Moreover, under these conditions there were practically no reversion products formed. Having obtained the inversion constant for maltose, mixtures containing known amounts of maltose and dextrose were made, the gravity of the solution of the mixed sugars was taken, and the grammes per 100 c.c. calculated by the 3·86 divisor. Ten c.c., or other appropriate volume, of the sugar solution was then weighed and the solid matter present calculated. The reducing power in terms of dextrose was determined before and after inversion. The percentage of maltose found was calculated from the formula:

$$M = \frac{(R - R_1)}{100 - 62{\cdot}1} \times 100,$$

in which R and R_1 are the reducing powers in terms of dextrose after and before inversion respectively. The constant 62·1 is the reducing power of anhydrous maltose, dextrose being taken as 100.

Approximate Percentage by Weight of added Maltose to Dextrose.	Reducing Power in terms of Dextrose before Inversion.	Reducing Power in terms of Dextrose after Inversion.	Percentage of Maltose found.
2·5	99·6	100·1	1·3
5·0	97·9	100·15	5·9
10·0	96·5	100·2	9·9
15·0	94·07	100·1	15·8

These results, which are the mean of many determinations, are fairly satisfactory, for it will be clear that a very small experimental error in the determination of the reducing powers will seriously affect the percentage of maltose.

If maltose and dextrose are the only sugars in the mixture which is being analysed, the inversion method will afford fairly accurate results; but should any other body be present, capable of forming reducing sugars on hydrolysis (dextrin, for example), the difference in the reducing powers before and after inversion would be calculated as maltose.

Since the number of reactions which can be utilized for the estimation of maltose in presence of dextrose is very limited, we decided to see if anything could be done by employing yeasts of selective fermentative functions. Hansen and other investigators have shown that certain yeasts, amongst them *Saccharomyces Marxianus* and *Ludwigii*, possess the property of fermenting dextrose and not maltose. We employed *S. Marxianus*. It was obviously necessary to ascertain (1) if the culture of *S. Marxianus* would have any reverting influence on dextrose, and (2) if a maltose hydrolyzing enzyme was present. Three hundred c.c. of a 1 per cent. solution of dextrose to which some yeast extract had been added were fermented for five days at 25° C. About 60 per cent. of the dextrose was fermented. The remaining sugar had the constants $(\alpha)_{D_{3\cdot86}} = 52\cdot1$ and $R_{3\cdot86} = 97\cdot8$ per cent. dextrose (corrected for added yeast extract). Two hundred c.c. of a 1 per cent. solution of maltose was treated with *S. Marxianus* under similar conditions. At the end of five days the solution, which had undergone no change in concentration, had the constants $(\alpha)_{D\,3\cdot93} = 134\cdot5$, and $R_{3\cdot93} = 101\cdot2$ per cent. of maltose.

The two possibilities, therefore, have no foundation, hence it was perfectly safe to use the culture of *S. Marxianus* for the removal of dextrose from a mixture of that sugar and maltose.

The method of experiment was as follows: To 100 c.c. of a 2 to 5 per cent. solution of the mixed sugars 5 c.c. of sterile yeast extract (prepared by boiling washed brewery yeast with water and filtering) were added, and the solution infected with a pure culture of *S. Marxianus* and allowed to ferment for five days at 25° C. At the end of this time the solution was treated with a little alumina cream, filtered, boiled to remove the alcohol, and its gravity, specific rotatory power, and reducing power in terms of dextrose determined.

The undermentioned results were obtained:

Before Fermentation. Constants of Mixtures of varying Proportions of Maltose and Dextrose.		After Fermentation.	
$(\alpha)_{D_{3\cdot86}}$	$R_{3\cdot86}$ glucose	$(\alpha)_{D_{3\cdot86}}$	$R_{3\cdot86}$ glucose
56·5	97·3	111·3	34·7
59·1	96·6	88·0	63·1
	(Fermented for sixty hours)		
56·4	97·4	82·0	70·0
	(Fermented for sixty hours)		
56·4	97·4	108·5	37·6

The increase in the specific rotatory power and the marked drop in the reducing power after fermentation may be regarded as evidence of the presence of maltose.

In order to confirm this deduction, the sugar remaining in solution was treated with phenylhydrazine acetate in the water-bath for three-quarters of an hour and the small amount of dextrosazone formed removed by filtration. Maltosazone separated from the filtrate in all the above experiments, and after it was recrystallized from a small quantity of boiling water it had the characteristic ribbon-like appearance of maltosazone and melted at 180° C. The melting-point is low for maltosazone, but it is well known that a small quantity of dextrosazone considerably depresses the melting-point of the former.

The conclusions that we have arrived at in this work may be summarized as follows:

1. L. Grimbert's method (*loc. cit.*) for removing the subsidiary products of the osazone reaction by benzene failed to give satisfactory results in our hands owing to the solubility of the impure maltosazone in this solvent. A small quantity of cold 5 per cent. aqueous acetone removes most of the impurities, without to any great extent dissolving the maltosazone. By relying on the osazone reaction alone, it was only possible to detect quantities of 15 per cent. maltose in mixtures of this sugar and dextrose with any degree of certainty.

2. Unsuccessful efforts were made to estimate small quantities of maltose in presence of dextrose by converting into osazones, and dissolving the maltosazone out with 20 per cent. acetone.

3. Small quantities of maltose, in a mixture of maltose and dextrose, may be estimated with a fair degree of accuracy by determining the reducing power before and after inversion under standard conditions, which are described. The difference in reducing powers may be due to maltose, but, in order to be certain on this point, the solution of the mixed sugars is fermented with *S. Marxianus*. A rise in the specific rotatory power and fall in the reducing power is additional evidence of the presence of maltose. To place the matter beyond doubt, the solution after fermentation is converted into maltosazone, and this compound identified as such by its microscopical appearance and melting-point.

Should dextrinous bodies of high molecular weight be present in the solution to be analysed, they must be removed before the inversion and fermentation. This may be done by repeated precipitations in alcohol, or mixtures of alcohol and acetone.

The methods described above are being used by the authors in an investigation on the products of the acid hydrolysis of starch.

DISCUSSION.

The PRESIDENT (Mr. Fairley) having invited discussion,

Dr. THORNE said that, when this set of carbohydrates occurred together in a mixture, even their identification, not to mention their determination, was a very difficult problem, towards the elucidation of which the author's work was certainly an important step. It seemed to him, however, that dextrin, if present, would cause a difficulty in the fermentation process, quite as great as that which, as stated in the paper, it caused in the reduction process. He must say that he had not found fermentation processes altogether satisfactory. In his experience, the supposition that a given yeast fermented a substance completely, or left another substance entirely unfermented, did not work out in practice to anything like the full degree.

The author's own experience in the two days' fermentation experiment showed that in that case the dextrose had not been completely fermented, and the maltose was not left absolutely intact. Although with mixtures of dextrose and maltose numbers could be obtained which were approximately correct, it seemed to him that, if dextrin were present, and especially if cane-sugar were also present, the interference of these products would make even the identification of small quantities of maltose very difficult, if not doubtful. Possibly the authors might have something to say with regard to the influence of dextrin and of cane-sugar, if present, on these fermentation results.

Mr. CHAPMAN said that this communication was of special interest, inasmuch as it represented the first fruits of a scheme which had been adopted by the Society with the object of fostering and promoting investigation in connection with analytical chemistry, and of securing the revision of processes which on the face of them seemed likely to be useful to practising chemists, but which for one reason or another could not be justifiably accepted without some further examination. If the future communications to the Society under this scheme were as useful and as well carried out as the one to which they had just listened they would have reason to congratulate themselves on having conceived the idea of such a scheme. Mr. Baker had remarked that he and his colleague had, to use his own expression, "gone beyond their commission"; but they would be very glad if others would also "go beyond their commissions" with as good results as the present authors had. So far as this paper was concerned, he (Mr. Chapman) must confess that he would have liked to have heard a little more about the acetone method, particularly as to its ability to detect, say, a small quantity of dextrose in the presence of a comparatively large quantity of maltose. Perhaps the authors had some further results bearing on this point. He thought there could be no doubt that the adoption of these three methods and a careful consideration of the results obtained would enable the one sugar to be estimated in presence of the other with a fair degree of accuracy. With regard, however, to the fermentation process, it must be borne in mind that the employment of a pure culture of any organism was apt in unpractised hands to be followed by misleading results. It was well known how prone such cultures were to become impure, and even a slight contamination might after a little time become serious. Those who were accustomed to working with pure cultures knew how to check their purity and to ascertain whether they had become contaminated, and they knew, also, what those unaccustomed to such matters might, perhaps, not know, namely, how necessary it was that such checking should be systematic. The detection of contamination was much more difficult than in the case of a chemical reagent, and could, practically speaking, only be carried out by those who were constantly handling such pure cultures. In short, the biological method was in many cases very useful; but it was apt to be dangerous in inexperienced hands. With regard to other points raised in the paper, he had found the reducing power of maltose in terms of dextrose to be distinctly lower than 62, and also that the melting-point of maltosazone was lower than the authors had found. He had purified maltosazone over and over again from every liquid from which it would crystallize, and had never been able to get the melting-point as high as 202°.

Mr. Baker, in reply, said that in the case of products consisting presumably of maltose, dextrose and dextrin, it was suggested that the dextrin should be first removed by precipitation with alcohol, or acetone, or a mixture of the two. With a mixture of maltose, dextrose, and cane-sugar, the cane-sugar should be inverted by yeast at about 55° C., and then fermented by *S. Marxianus*. In response to Mr. Chapman's desire for further information about the treatment of the osazones with acetone, he might say that after some experiments it had been found that dextrosazone was very appreciably soluble in 50 per cent. acetone, and by a process of reduction it was found that much better results were obtained with 20 per cent. acetone, in which dextrosazone was practically insoluble. Mixtures of maltose and dextrose had been made containing 2½, 5, 10 and 15 per cent. of maltose, and it was found that, extracting with 20 per cent. acetone, a mixture of 10 or 15 per cent. of maltose in presence of dextrose could be detected, but not 2½ or 5 per cent. When the osazones were first isolated and then mixed, these smaller quantities could be detected, but it was not possible when the sugars themselves were mixed, owing to the formation of subsidiary reaction products. Another point to which attention might be drawn was that in the original method of Grimbert the benzene used removed practically all the maltosazone when this substance was present in small amount. With 15 or 10 per cent. of maltose it did not matter, but with 2½ or 5 per cent. the method could not be used. Mr. Chapman's remarks on the use of biological reagents were perfectly just, but, as had been pointed out, practically no strong evidence of the presence of the maltose was to be obtained by inversion, and some other method was absolutely necessary. The only one applicable in the present instance, so far as he was aware, was the use of a biological reagent—namely, *S. Marxianus*. In sugar analysis it seemed that such unusual reagents must sometimes be resorted to, but he quite agreed that it was well to recognise their danger in unaccustomed hands. The melting-point of maltosazone depended a good deal upon how the determination was made. If the sulphuric acid was heated very slowly, the maltosazone invariably melted at about 190° C. If it were heated quickly, according to Fischer's instructions, pure maltosazone would melt at 195° to 200° C. His own practice was to take the melting-point when the sintered mass ran together, and under those conditions he found the melting-point of pure maltosazone to be 195° to 200°, and that of dextrosazone 202° C.

Mr. Chapman: With dextrosazone it is much sharper than with maltosazone?

Mr. Baker: Yes.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Foreign Fat in Milk. G. Quesneville. (*Ann. de Chim. anal.*, 1904, ix., 457, 458.)—Assuming the correctness of the theory that the fat globules in milk are surrounded by an albuminoid envelope, the author has looked for a solvent that would dissolve fat without acting upon this albuminoid envelope, and has found benzene (boiling-point, 81° C.) suitable for the purpose. On shaking the cream (80 to 110 c.c.) separated from a litre of milk with 100 c.c. of benzene purified by recrystallization, and evaporating 50 to 80 c.c. of the extract, a residue corresponding to 0·2 to 0·5 gramme (at most 1 gramme) of fat will be obtained from a litre of milk containing 40 to 45 grammes of butter fat. If, on the other hand, the cream from a milk in which 15 grammes of butter fat have been replaced by 15 grammes of foreign fat be treated in the same way, a large residue of fat will be left. Thus, in one experiment in which the cream was shaken with 150 c.c. of benzene, 110 c.c. of the clear supernatant layer evaporated, and the residue dried at 90° to 95° C.; the extracted fat weighed 9 grammes, corresponding to a total amount of 13 grammes dissolved by the 150 c.c. of the benzene. Similar results were obtained with boiled milk, not more than 1 gramme of fat per litre of milk being extracted by benzene from the cream. C. A. M.

A Simple Method for the Determination of the Fat in Butter. A. Hesse. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 673-675.)—The method is a modification of Gottlieb's process for the determination of fat in milk (ANALYST, 1898, 259). From 1·5 to 2 grammes of the well-mixed sample are weighed out into a stoppered cylinder and melted by the addition of about 8 c.c. of hot water. If necessary, the cylinder may be placed in hot water to complete the melting. One c.c. of ammonia and 10 c.c. of alcohol are now added, and the cylinder shaken until the proteids have dissolved. After cooling, the mixture is first shaken with 25 c.c. of ether and then with 25 c.c. of petroleum spirit. As soon as separation has taken place, the ethereal layer is pipetted off, 50 c.c. of ether are added to the cylinder, and pipetted off without shaking up. The contents of the cylinder are finally shaken with 50 c.c. of a mixture of equal parts of ether and petroleum spirit, and the ethereal layer is once more removed with a pipette. The united ethereal solutions are evaporated, and the residue of fat weighed. It is claimed that the latter contains no non-fatty matters. From the results given the method appears to be trustworthy W. P. S.

The Molecular Weight of the Non-volatile Fatty Acids of Dutch Butter. A. Olig and J. Tillmans. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 728-730.)—The results of analyses of Dutch butters, churned from the milk by the authors themselves, are given, showing that in the summer the molecular weight of the non-volatile fatty acids generally, but not always, lies within the limits mentioned by Juckenack and Pasternack (ANALYST, 1904, 156). In the autumn, however, the molecular weights were often above 261·0, the highest value obtained being 271·6. The results show, further, that this determination is not by itself a trustworthy method of detecting adulteration in butter fat. W. P. S.

The Cryoscopic Method for Distinction of Genuine from Artificial Butters. A. Quartaroli. (*Staz. Sperim. Agraria Ital.*, 1904, xxxvii., 18; through *Chem. Zeit. Rep.*, 1904, xxviii., 354.)—The author uses glacial acetic acid in place of the benzene recommended by Garelli for this method.

Five to six grammes of dried filtered butter are shaken with 50 c.c. of glacial acetic acid, and allowed to stand for twenty-four hours. The solution is then filtered and the depression of the freezing-point determined. With fresh genuine butter the depression was 0·54 to 0·57 per cent.; old samples gave 0·52 to 0·53 per cent., while margarine showed only 0·17 to 0·20 per cent. A mixture of 20 per cent. of margarine in butter reduced the freezing-point 0·36 per cent. H. A. T.

The Composition of Fresh Almonds. F. Coreil. (*Ann. de Chim. anal.*, 1905, x., 21-23.)—Three specimens of the green fruit had the following composition:

	Hard. Per Cent.	Semi-hard. Per Cent.	Soft. Per Cent.
Bark	87·00	83·00	81·10
Tegument of the nut	3·20	4·60	3·55
Edible part (cotyledons) ...	9·80	12·40	15·35

The edible part consisted of the following constituents:

	Per Cent.	Per Cent.	Per Cent.
Water	7·53	10·006	11·61
Solids	2·27	2·394	3·74
Fat	1·247	1·370	2·109
Nitrogenous substances	0·077	0·090	0·133
Mineral matter	0·142	0·158	0·210
Cellulose and carbohydrates ...	0·804	0·776	1·288
Phosphoric acid	0·038	0·046	0·059

C. A. M.

The Influence of Ozone on Wheat Flour. K. Brahm. (*Zeit. Untersuch. Nahr. Genussmittel*, 1904, viii., 669-673.)—As ozone, or ozonised air, is occasionally used to bleach wheat flour, the author carried out a number of experiments, in which he determined the alterations which flour undergoes when thus treated. When allowed to act for less than one hour, the ozonised air produced but little change in the physical and chemical characters of the flour, but imparted an "ozone taste" to it, which did not disappear on heating the flour to 100° C. Prolonged bleaching (several hours) considerably decreased the quantity of gluten yielded by the flour, although the total nitrogen remained the same, showing that it was only the physical properties of the gluten which had been altered. This was confirmed by mixing the flour, both treated and untreated, with yeast, water, salt, etc., as in bread-making, and allowing the dough to "rise" at a temperature of 30° C. The flour which had been strongly bleached had very little "rising" power. Bread made from ozonised flour was poor in appearance, of a yellow colour, pasty consistency, and unpleasant taste. Bleaching by ozonised air is therefore considered to have an injurious effect on the flour as regards the suitability of the latter for bread-making. W. P. S.

Determination of Commercial Glucose in some Saccharine Products.

A. E. Leach. (*Massachusetts State Board of Health, 35th Annual Rep.*, 1903, 60, 61.)—If the invert polarization at 87° C. of a sample of honey, jam, molasses, or syrup be divided by the assumed factor for the polarization of commercial glucose, the percentage of the latter is obtained. Several precautions are, however, to be considered in carrying out the method. The direct polarization is obtained on a normal solution of the sample in the usual manner. For the invert reading a second solution is prepared by dissolving 13·024 grammes of the sample in about 70 c.c. of water, adding 7 c.c. of concentrated hydrochloric acid, heating the mixture to 68° C., and cooling. A few drops of phenolphthalein are then added and enough sodium hydroxide to neutralize the solution. This neutralization is necessary to prevent inversion of the maltose and dextrin in the glucose at the high temperature of reading. After discharging the pink coloration by the addition of a drop of dilute hydrochloric acid, the usual clarifying agent is added and the whole made up to a volume of 100 c.c. Readings of this solution are taken at 22° and 87° C. respectively. The following table shows the readings obtained with five samples of commercial (American) glucose:

No.	Density.	A. Direct Reading.	B. Invert Reading at 22° C.	C. Invert Reading at 87° C.	Ratio of C to B.	Ratio of C to A.
1	42° B.	156·6	153·4	146·6	0·956	0·936
2	42° B.	158·6	154·6	149·0	0·964	0·940
3	42° B.	169·6	165·4	159·4	0·964	0·940
4	43° B.	167·4	162·8	155·0	0·952	0·926
5	45° B.	174·0	171·0	161·2	0·943	0·927
			Average	... ...	0·956	0·933

It will therefore be necessary in the case of jam, etc., to divide the reading at 87° C. by 93 per cent. of the assumed polarizing value of commercial glucose (for example, 175)—that is, by 163—in order to express the results in terms of the latter.

W. P. S.

The Detection and Estimation of Citric Acid in Wines.

L. Robin. (*Ann. de Chim. anal.*, 1904, ix., 453-456.)—Twenty-five c.c. of the wine are heated to the boiling-point, mixed with 3 c.c. of a 40 per cent. solution of neutral lead acetate, and the boiling continued for one or two minutes. The precipitate is then allowed to subside, and the liquid filtered while still hot. The precipitate, consisting of lead sulphate, phosphate, tartrate, and citrate, is washed with warm water, and heated for five minutes at 90° C. with 10 c.c. of water acidified with acetic acid.

The liquid is decanted on to a filter, and the filtrate and washings containing the tartrate and citrate of lead are saturated with hydrogen sulphide and filtered. This filtrate is evaporated nearly to dryness in a porcelain dish, the residue dissolved in 5 c.c. of 90 per cent. alcohol, and the solution exactly neutralized with potassium hydroxide, and then acidified with 0·5 c.c. of glacial acetic acid, and thoroughly mixed. The liquid is transferred to a small flask, and the dish washed twice with 5 c.c. of 90 per cent. alcohol and 0·5 c.c. of glacial acetic acid. The flask is left for an hour in a cool place, during which time the potassium bitartrate will have been deposited, while the citrate is kept in solution by the acetic acid. The liquid is filtered, the filter washed with acidified alcohol, and the filtrate shaken with 5 or 6 drops of a saturated solution of cadmium acetate. If citric acid had been added to the wine a flocculent precipitate is obtained. For a quantitative determination, the cadmium citrate is collected on a weighed filter, washed with 90 per cent. alcohol, and dried for two hours at 100° C. The weight multiplied by 0·5738 and then by 40 gives the amount of citric acid in a litre of the wine. Denigès' qualitative test (ANALYST, xxix., 118) has not given satisfactory results in the author's hands. C. A. M.

The Nitrogenous Constituents of Flesh. H. S. Grindley. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1086.)—The elaborate results recorded in this paper are a preliminary step towards the elucidation of the chemistry of the proteids as they exist in meat used for food. A uniform method of analysis was followed, the meats being successively extracted with the following liquids in the order named: Cold water (up to 10° C.); 10 per cent. sodium chloride solution; 0·15 per cent. hydrochloric acid; 0·15 per cent. caustic potash solution; and boiling water. In each solution determinations of the total nitrogen, coagulable proteid, albumoses (by precipitation with zinc sulphate), peptones (by precipitation with bromine water), and flesh bases, were made. After the extraction treatments with these solvents the following amounts of insoluble proteid matter were found in the final residues: Lean beef, raw, 0·71 and 1·34 per cent.; lean beef, boiled, 18·69 per cent.

The author calls attention to the fact (previously noticed by Van Slyke and others) that loss of nitrogen occurs in using bromine as a precipitant for peptones, since this reagent decomposes the flesh bases and similar nitrogenous bodies, with liberation of free nitrogen. It is also found that bromine does not precipitate all the proteids completely from a solution.

The cold-water extracts of raw flesh invariably contain a small amount of nitrogen existing in the form of ammonia or ammonium salts, even after previous removal of these by distillation with barium carbonate.

As a result of his experiments, the author draws the following conclusions:

1. It is evident that a considerable proportion of raw flesh is soluble in cold water.

2. The data show that 12·14 per cent. of the total proteids of raw flesh were soluble in cold water.

3. Of the total nitrogen in raw meats, 22 per cent. was soluble in water.

4. The nitrogen existing in cold water extracts of raw meat is equally divided between proteid and non-proteid substances.

5. The acidity of a solution of flesh increases upon the coagulation of its proteids.

6. The proteids of cooked meat are much less soluble than those of raw flesh in cold water and 10 per cent. sodium chloride solution.

7. Cold water extracted 3·06 per cent. of nitrogenous matter from raw meats and only 0·27 per cent. from boiled meat.

8. A 10 per cent. solution of sodium chloride extracted from raw meats 6·10 per cent. of proteid matter, and only 0·5 per cent. from boiled meat.

9. A 0·15 per cent. solution of hydrochloric acid dissolved from raw meat 2·28 per cent. of proteid, and from boiled meat 2·30 per cent.

10. A 0·15 per cent. solution of caustic potash extracted from raw meats 2·88 per cent., and from boiled meat 4·84 per cent. of proteid.

11. Hot water removed from raw meats 0·49 per cent., and from boiled meat 6·24 per cent. of proteid matter.

12. Of the total proteid existing in the original raw meats, 95·22 per cent. was dissolved by successive treatment with the above-mentioned reagents, while only 50·59 per cent. of the total proteid of the boiled meat was soluble.

The paper is illustrated by a large number of tables of analyses, for which the original must be consulted. The author hopes to publish shortly the results of his work on the separation and purification of the different proteids of flesh.

A. R. T.

The Solubility of Different Caseins in Pepsin-Hydrochloric Acid. A. Zaitschek and F. v. Szontagh. (*Pflüg. Arch.*, 1904, civ., 550-563; *Biochem. Centralbl.*, 1905, iii., 411, 412.)—In a series of parallel digestions for seventy-two hours at 38° C. it was found that the crude caseins of human milk and of mare's and ass's milk were entirely dissolved by pepsin-hydrochloric acid, whilst only 8 per cent. of cow's milk, 14 per cent. of buffalo's milk, and 15 per cent. of goat's milk were respectively digested under the same conditions. These results were also confirmed in experiments with the purified caseins.

C. A. M.

Determination of Sulphur and Phosphoric Acid in Foods, etc. J. A. LeClerc and W. L. Dubois. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1108.)—The following methods were used for the determination of these substances in foods, fæces, and urine, in connection with the experiments of Dr. Wiley and Dr. Bigelow on the influence of preservatives in food on the human body.

Determination of Sulphur and Phosphorus in Foods and Fæces.—(1) About 1 to 1¼ grammes of the dried and finely-ground sample is placed in the cartridge of the Parr calorimeter (*Journ. Amer. Chem. Soc.*, xxii., 646), 10 to 15 grammes of sodium peroxide added, and the cartridge closed and the contents thoroughly mixed by shaking. The mixture is ignited by means of a red-hot iron wire introduced through the valve, the cartridge being meanwhile immersed in cold water up to its narrow part. Combustion takes place immediately, and, after cooling, the contents of the cartridge are dissolved in hot water, acidified with hydrochloric acid, and the excess of sodium peroxide decomposed by boiling. In the case of foods, the filtered liquid is made up to 200 c.c., and 50 c.c. of the solution taken for the phosphoric acid determination (see (3) below); the remainder is precipitated with barium chloride. In the analysis of fæces, the liquid, after acidification, is made up to 300 c.c., and 50 c.c. taken for the determination of phosphoric acid (3). The remainder of the solution is treated with ammonia, to remove the phosphates, before determining the sulphate. This precaution is advisable, since phosphates are otherwise liable to become occluded with the barium sulphate.

2. The Osborne method is tedious and somewhat uncertain. To 10 c.c. of water in a nickel crucible of 100 c.c. capacity are added about 10 to 12 grammes of sodium peroxide, avoiding excess, and then 4 grammes of the meat or other fresh food, or 2 grammes of dried food or fæces. The water is largely evaporated on a water-bath, and the mixture then heated over a low flame, increasing the heat cautiously. Much frothing takes place, and the froth should not become ignited. The heat is further increased, when the frothing has subsided, until the mass begins to fuse, when, after cooling, a little more sodium peroxide is added and the mixture re-heated. This is continued until no more action takes place, and oxidation is complete. The mass is then dissolved, boiled, and diluted, etc., as already described in (1).

The Parr calorimeter method would be preferable, but the cartridge is so small that sufficient sample cannot be taken. One holding 2 to 3 grammes is desirable, and a more practicable process than that of Osborne would then be available.

3. For the determination of phosphoric acid in the liquid obtained by either of the foregoing processes, the official method (*Bureau Chemistry, Dept. of Agric., Bull.* xlvi., revised, p. 14) is employed, except that the solution is precipitated cold, and allowed to stand overnight at the ordinary temperature. The precipitate is finally washed until no trace of acid is present, when it is titrated as usual.

Determination of Sulphur and Phosphorus in Urine.—(*a*). The sulphur is determined by Osborne's sodium peroxide method, already described. A little less water and peroxide than before and 20 c.c. of urine are employed, and the mixture evaporated on the water-bath. The mass is then gently heated, and the method subsequently continued as in the case of foods, etc. (2). Very little frothing should occur.

(*b*) The phosphoric acid in urine is determined by the usual titration method with uranium acetate, using potassium ferrocyanide as an outside indicator. Fifty c.c. of urine are diluted to 150 c.c. with water before heating and titrating. Undiluted urine was found to give low results. A. R. T.

Detection of Artificial Colouring Matters in Mustard. W. Schmitz-Dumont. (*Zeit. öffentl. Chem.*, 1904, x., 487.)—While agreeing that the tests given by Bohrisch (ANALYST, 1904, 372) are usually sufficient to detect the presence of aniline colours in mustard, the author points out that samples coloured with dyes of the tropæolin type behave like genuine mustard when tested by Bohrisch's method. The dyed wool should always be moistened with hydrochloric acid, when, if tropæolins be present, the brownish-yellow colour of the wool changes to bright red or violet. W. P. S.

Determination of the Cold-Water Extract yielded by Ground Ginger. A. McGill. (*Canadian Inland Revenue Dept. Bull.*, xcv., 24, 25.)—The following method is described, and has for its main feature the mechanical agitation of the ginger with the solvent. Five grammes of the sample are dried in the water-oven until constant in weight, which usually takes about twelve hours or more. The dry sample is then transferred by means of a dry funnel to a centrifugal tube of about 150 c.c. capacity and treated with 100 c.c. of water. After closing, the tube is placed in a horizontal shaking apparatus for thirty minutes and then in a centrifugal machine for ten minutes. The clear supernatant liquid is poured through a tared filter, and the insoluble residue again treated with 100 c.c. of water, shaken, whirled, and the clear solution passed through the filter. Finally, the residue is washed on the filter with about 100 c.c. of water, the exact quantity being immaterial, as only traces of soluble matter remain in the residue. The filter and its contents are dried at a temperature below 50° C. for a few hours, and afterwards at 90° to 95° C. until dry. Genuine samples of ground ginger yield about 19 per cent. of cold-water extract by this method, an amount which is considerably higher than that obtained by the usual method of extraction. W. P. S.

Flowers of Sulphur and Sublimed Sulphur. A. Domergue. (*Journ. Pharm. Chim.*, 1904, xx., 493-499.)—The condensed vapours of sulphur are known indifferently as either *flowers of sulphur* or *sublimed sulphur*, but in the author's opinion there should be a differentiation between the two names for commercial products. At the commencement of the condensation the difference in temperature between the sulphur vapour and the atmosphere of the chamber leads to the sudden formation of a globular form of deposit, whilst at a later stage the powder is crystalline. The author suggests that the former should be termed *flowers of sulphur*, whilst sulphur containing variable proportions of the crystalline and globular forms should be sold as *sublimed sulphur*. The microscope affords a means of distinguishing between the two qualitatively, and a quantitative test may be based on the proportion insoluble in carbon bisulphide. Various commercial products examined by the author contained from 13 to 33 per cent. of insoluble sulphur, while samples taken direct from the chamber contained from 25 to 37 per cent. The conclusion arrived at by the author from these results is that the name *flowers of sulphur* ought to be reserved in commerce for those products giving at least 33 per cent. of sulphur insoluble in carbon bisulphide at the time of manufacture, whilst other products of the condensation chamber should be termed *sublimed sulphur*. C. A. M.

Physical Characteristics of Adrenaline. G. Bertrand. (*Bull. Soc. Chim.*, 1904, xxxi., 1289-1292.)—Pure adrenaline forms crystals arranged in rosettes round common centres. It is only slightly soluble in water. At 20° C. the amount dissolved is 0·0268 per cent., whilst at 100° C. its solubility is somewhat greater. It is still less soluble in alcohol, and is insoluble in ether, chloroform, carbon bisulphide, and petroleum spirit. Adrenaline is optically active, the mean rotation found by the author being $[\alpha]_D = -53·5°$. It does not show a sharp melting-point. When thrown in a fine powder on to a Maquenne's block it only melted instantly when the temperature was about 263° C., and the same result was obtained after further purification of the alkaloid (*cf.* ANALYST, xxx., 22). C. A. M.

New Colour Reactions of Cryogenin. G. Pégurier. (*Ann. de Chim. anal.*, 1904, ix., 456, 457.)—(1) Cryogenin (*m*-benzamido-semicarbazide) gives a chestnut-brown coloration with potassium permanganate. (2) On heating a few dry crystals of cryogenin in a test-tube over a spirit-lamp a brownish-black coloration is produced, whilst the liberated vapours have a strong ammoniacal odour. (3) A brilliant garnet-red coloration is obtained on treating crystals of cryogenin with a drop of pure sulphuric acid and a drop of potassium bichromate solution. C. A. M.

Characters of Santalol and Sandal-wood Oil. Seidler. (*Apoth. Zeit.*, 1904, xix., 795; through *Pharm. Journ.*, 1904, lxxiii., 813.)—Santalol, the active constituent of sandal-wood oil, has the following characters: Specific gravity, at least 0·980; optical rotation in 100-millimetre tube, -18° to -19°. It forms a clear solution with 4 parts of alcohol (70 per cent. by volume). Santalol distils almost entirely between 302° and 306°. On acetylation, the product should require a definite quantity of caustic potash for saponification.

Pure East Indian sandal-wood oil has a pale yellow colour, and only a slight odour of sandal-wood. Its specific gravity is 0·975 to 0·985, and optical rotation -17° to -19° for 100 millimetres. The oil dissolves in 5 parts of 70 per cent. (volume) alcohol. The acid number, ester, and saponification numbers do not exceed, in pure samples, the limits 2·75, 5 (at the most), and 7·5 respectively. The saponification number of the acetylated oil is at least 197. The oil distils mainly between 297° and 305°. A. R. T.

Composition of Pine Oils. J. Tröger and A. Bentin. (*Archiv. der Pharm.*, ccxlii., 521; through *Pharm. Journ.*, 1904, lxxiii., 900.)—The oils distilled from the young shoots of *Pinus sylvestris* and *Pinus strobus* contain dextro-pinene and lævo-pinene respectively in large proportion, but are free from sylvestrene, cadinene, and the borneol esters which are present in the oils from the older leaves. These bodies are therefore produced during the later growth of the leaves. A. R. T.

Fixed Oil of Carthamus Tinctorius Fruit. G. Fendler. (*Apoth. Zeit.*, xix., 721; through *Pharm. Journ.*, 1904, lxxiii., 900.)—This fruit, obtained from German East Africa, yields 25·82 per cent. of fixed oil. The separated kernels of the seeds yield 50·37 per cent. of a slow-drying oil having the following characters: Specific gravity at 15° C., 0·9266; melting-point, +5° C.; Reichert-Meissl number, 0; acid number, 11·63; saponification number, 191; iodine value, 142·2; and refractometer reading at 40° C., 65°. The oil becomes cloudy at -13°, and is not wholly solid at -18° C. The fatty acids of this oil have the following characters: Specific gravity at 15° C., 0·9135; melting-point, 17° C.; freezing-point, 12° C.; acid number, 199; mean molecular weight, 281·8; acetyl number, 52·9; and iodine value, 148·2. The liquid fatty acids have an iodine value of 150·8, and a mean molecular weight of 293·1. A. R. T.

TOXICOLOGICAL ANALYSIS.

The Detection of Poisons in the Ashes left after Cremation. C. Mai and H. Hurt. (*Zeit. angew. Chem.*, 1904, xxvii., 1601-1605.)—Experiments were carried out to ascertain whether arsenic, mercury, or cyanogen compounds could be detected in the ashes left after cremating the bodies of animals which had been poisoned with these substances. Dogs and rabbits were employed, and the bodies were burnt in a brick furnace heated by coal. The latter was practically free from arsenic. Arsenic was readily detected in the ashes, the greater quantity being found in the bones. Mercury salts, such as the perchloride and cyanide, completely disappeared during the cremation, and the ashes were free from this metal. With regard to cyanogen, it was found that cyanogen compounds are not formed when flesh is burnt, and, further, that these compounds cannot be detected in the ashes of animals poisoned with potassium cyanide or prussic acid. W. P. S.

On the Presence of Arsenic in the Body and its Secretion by the Kidney. W. Thomson. (*Proceedings Manchester Literary and Philosophical Society*, 1904-5, xlix., 1, 10.)—Having found that the urine of people living in Manchester contained arsenic, the author extended his researches in order to discover, if possible, the source of the contamination, and, further, to ascertain how rapidly arsenic is eliminated from the body. Six samples of urine from patients in the Manchester Southern Hospital all contained arsenic, the quantity varying from $\frac{1}{1000}$ to $\frac{1}{100}$ grain per gallon. The latter quantity was in the urine of a woman who had been in hospital thirteen days, and who had taken no medicine during that time. Five samples from people living at St. Helens, where large quantities of arsenic escape into the atmosphere from the glassworks, contained from $\frac{1}{400}$ to $\frac{1}{300}$ grain of arsenic per gallon. The quantities present in six samples from Swansea varied from $\frac{1}{700}$ to $\frac{1}{80}$ grain per gallon, and in five samples from Sheffield from $\frac{1}{1000}$ to $\frac{1}{100}$ grain per gallon. Samples obtained from St. Anne's-on-the-Sea and the Hebrides, where the air was not likely to be contaminated, were either quite free from arsenic or practically so. Six samples of milk purchased in or near Manchester all contained arsenic, the quantity present ranging from less than $\frac{1}{1000}$ to $\frac{1}{450}$ grain per gallon. A piece of beefsteak grilled over a coke fire was found to contain not more than $\frac{1}{2000}$ grain of arsenic per pound even in the outside portions. Four different brands of cigarette-paper were next examined. All were arsenical, the amounts varying from $\frac{1}{1000}$ to $\frac{1}{250}$ grain per pound.

With regard to the elimination of arsenic by the kidneys, an experiment was carried out in which $\frac{1}{50}$ grain of arsenious acid was administered to a man. Previous tests showed the quantity of arsenic in his urine to be $\frac{1}{1000}$ grain per gallon. All the urine passed was collected and tested. Seven hours after taking the dose the amount of arsenic had risen to $\frac{1}{30}$ grain per gallon. At the end of twenty-three hours about 16 per cent. of the quantity taken had been secreted, and after forty-seven hours the urine contained only $\frac{1}{1000}$ grain per gallon, which was the quantity present previous to making the experiment.

A human thyroid gland, weighing about 18 grammes, was found to contain only a minute quantity of arsenic, $\frac{1}{1500}$ grain per pound, so that arsenic can scarcely be spoken of as a constituent part of this gland, as it has been stated to be by Gautier.

The spleen, liver, heart-muscle, kidney, and rib-bone of the same body were free from arsenic, but the lung contained $\frac{1}{500}$ grain per pound, and the hair $\frac{1}{125}$ grain per pound. W. P. S.

ORGANIC ANALYSIS.

Colour Reactions of Certain Alcohols and Hydroxylated Bodies.

G. Guérin. (*Journ. Pharm. Chim.*, 1905, xxi., 14-17.)—One c.c. of the alcohol under examination is mixed with 5 or 6 drops of a saturated aqueous solution of furfural, and the mixture shaken with an equal volume of concentrated sulphuric acid. In the case of solid substances, a little is dissolved in sulphuric acid or in ethyl alcohol, and the solution cautiously treated as described. Or a little of the substance may be mixed on white porcelain with 4 or 5 drops of sulphuric acid and a drop of the aqueous solution of furfural. No colour reactions are given by methyl or ethyl alcohols, and a brown or brownish-violet coloration is simply due to the action of the sulphuric acid on the furfural, and may be neglected. The following colorations were obtained in this test: Normal propyl alcohol, deep violet; secondary propyl alcohol, reddish violet; isobutyl alcohol, violet blue; active amyl alcohol (methyl-ethyl-carbinol), violet; amyl alcohol of fermentation, violet; caprylic alcohol, reddish violet; glycol, reddish violet; propyl-glycol, violet purple; glycerin, reddish violet; allyl alcohol, immediate brown coloration preceding almost immediate decomposition; menthol (in ethyl alcohol solution), blue; mannitol (dissolved in sulphuric acid), brownish green—in ethyl alcohol, brownish violet; dextrose (in sulphuric acid or ethyl alcohol), brownish violet; lactose, brownish; malic acid, brownish violet; tartaric acid, brownish violet; cholesterol, blue (sensitive test); anthemol (from *Anthemis nobilis*), blue; phenol, reddish violet; thymol, violet; guaiacol (in ethyl alcohol), violet; orcinol, blue; hydroquinone, dark blue; pyrocatechol, deep violet; resorcinol, violet; phloroglucinol, violet; pyrogallol (in ethyl alcohol), reddish violet. C. A. M.

The Volumetric Determination of Hydroxylamine by means of Trivalent Titanium.

Stähler. (*Berichte*, 1904, xxxvii., 4732, 4733.)—The method is based on the reduction of the hydroxylamine to ammonia:

$$Ti_2O_3 + NH_3O = 2TiO_2 + NH_3.$$

Titanium tetrachloride or sulphate is dissolved in sulphuric acid and reduced by means of nascent hydrogen until the solution attains the required reducing power, which may be determined by titration with $\frac{N}{10}$ potassium permanganate solution. During the final standardization with the permanganate solution a current of carbon dioxide is passed through the flask. In the determination 4 grammes of hydroxylamine sulphate are dissolved in 100 c.c. of water, 21 c.c. of the solution, diluted with boiling water, then rapidly mixed with an excess of the standard titanium solution, and the excess of titanium trichloride determined by titration with $\frac{N}{10}$ permanganate solution in a current of carbon dioxide. The method is also applicable to the determination of organic hydroxylamine compounds, whilst hydrazine sulphate is not altered by titration with titanium trichloride under the same conditions.

C. A. M.

A Reaction of Sugars containing the Aldehydic Group. A. Berg.

(*Bull. Soc. Chim.*, 1904, xxxi., 1216-1217.)—Bromine in the presence of water oxidizes aldoses, converting the aldehydic into an acid group, with the formation of acids, such as gluconic, galactonic acids, etc. These acids give an intense yellow coloration with very dilute and slightly acid solutions of ferric chloride. On the other hand, ketoses are not converted into acids under the regulated action of bromine. In examining a sugar, from 0·02 to 0·03 gramme in the solid state or in concentrated solution is heated for ten minutes at 60° to 70° C. with 10 c.c. of a freshly-prepared saturated solution of bromine. The liquid is then rapidly heated to the boiling-point to expel the excess of bromine, and the colourless solution tested with 10 c.c. of a reagent prepared by adding to 100 c.c. of water 4 drops of a solution of ferric chloride (45° Bé., S.G. 1·45), and 2 drops of hydrochloric acid (22° Bé., S.G. 1·17). Sugars not containing the aldehydic group give little or no coloration, and the slight coloration given by fructose and sorbose is to be attributed to the presence of traces of aldoses, which it is difficult to completely eliminate. A strong reaction is given by arabinose, xylose, glucose, and galactose. Of the complex sugars, bioses or trioses, those that do not reduce Fehling's solution give no reaction—*e.g.*, saccharose, raffinose, trehalose, etc.—whilst those with reducing properties also give marked colorations. In the case of polyoses it is necessary to use bromine water free from mineral acids—notably hydrobromic acid—in order to prevent inversion during the test, and it is for this reason that the bromine water should be freshly prepared from pure bromine. Commercial saccharose invariably gives a more or less pronounced yellow coloration, due to the presence of a little glucose. The latter can be removed by recrystallizing the sugar several times from boiling alcohol, after which it no longer gives the reaction. C. A. M.

Determination of Nicotine in Presence of Pyridine. James A. Emery.

(*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1113).—The author determines the proportion of total volatile bases in nicotine preparations by Kissling's process, and the amount of nicotine by the observation of the rotatory power of the solution. The difference gives the proportion of pyridine present, since the latter compound is optically inactive.

Five grammes of tobacco extract, or 20 grammes of dry, finely-powdered tobacco are treated with 10 c.c. of a solution containing 6 grammes of caustic soda in 60 c.c. of 90 per cent. alcohol and 40 c.c. of water. Tobacco extracts are, in addition, mixed with sufficient pure dry calcium carbonate to form a moist, but not lumpy, mass. After thorough mixing, the whole is transferred to a Soxhlet apparatus, and extracted with ether for five hours. The solvent is next evaporated on a water-bath at a low temperature, care being taken that the last portions of the ether are not heated sufficiently to cause volatilization of the nicotine or pyridine.

Fifty c.c. of decinormal caustic soda are now added to the contents of the flask, and the solution transferred to a distillation-flask (of 300 c.c. capacity) by means of

150 c.c. of water. The liquid is then distilled in a current of steam, in such a manner that when about 450 c.c. of distillate are obtained, only 10 to 20 c.c. of liquid remains in the distilling-flask. All the nicotine will then have passed over. The vapours should pass through a well-cooled condenser, the outlet-tube of which dips into a little water in the receiver. Bumping and frothing in the distillation-flask are avoided by the addition of a little pumice-stone and a small piece of paraffin-wax. The distillate is diluted to 500 c.c. with water, and an aliquot part titrated with fifth-normal acid and methyl-orange. This is essentially Kissling's process for the determination of the total volatile bases. Another portion of the distillate is placed in a polarimeter-tube, and its rotatory power determined. A 1 per cent. solution of pure nicotine is also prepared, and its rotation noted (the specific rotation of pure nicotine at 20° C. is $-161{\cdot}55°$); whilst it is also titrated to determine accurately its exact strength. Thus the value of one degree on the polarimeter scale can be ascertained in terms of percentage of nicotine, and if the percentage found be multiplied by 5, the amount of nicotine in the whole of the distillate will be obtained. If, then, the nicotine found be expressed in terms of standard acid, and the number of c.c. be subtracted from the volume of standard acid required to neutralize the entire distillate, a fairly accurate determination of the pyridine present can be made.

The following rapid method, for the determination of nicotine only, avoids the extraction with ether: A known weight of the sample is made alkaline with 50 c.c. of decinormal caustic soda, and distilled exactly as already described, after dilution with 150 c.c. of water. The nicotine in the distillate is then determined by the polarimeter. This method gives good results generally, except that in tobacco-powders slightly low results are obtained, owing to the difficulty of sufficiently reducing the volume of liquid in the distilling-flask without causing bumping of the wet powder in the flask.

The test-analyses given are very satisfactory. A. R. T.

On the Separation of Fatty Acids. W. Fahrion.

(*Zeit. angew. Chem.*, 1904, xvii., 1482-1488.)—It is here shown by experiments described in detail that Partheil and Ferié's method of separating saturated and unsaturated fatty acids (Analyst, xxix., 51) is unreliable. Although the lithium salts of unsaturated acids by themselves are completely soluble in 50 per cent. alcohol, yet in the presence of the lithium salts of saturated fatty acids (including lithium acetate) they become partially insoluble. On the other hand, the lithium salts of saturated acids are by no means insoluble in 50 per cent. alcohol, and their insolubility is still further reduced by the presence of lithium salts of unsaturated acids. Hence, in the precipitation of a mixture of fatty acids a condition of equilibrium results, to which the relative quantities and molecular weights of the different acids, the amount of precipitant, the concentration of the solution, and the temperature all contribute.

C. A. M.

The Detection of Rosin and Fish Oils in Boiled Linseed Oil. W. Lippert.

(*Chem. Rev. Fettu. Harz Ind.*, 1905, xii., 4, 5.)—Linseed oil boiled with lead or manganese oxides gives only brown colorations in Storch and Morawski's test, whilst red or blue colorations are obtained when resinates, rosin, or fish oils are present.

Maize oil also gives a similar reaction. Four years ago the high price of linseed oil led to extensive adulterations, and oil expressed from heads of sardines was largely exported from the coast of Spain for this purpose. Samples of such oil examined by the author gave the Storch-Morawski and other reactions for fish oil. After exposure of the oils to the light, the reactions were fainter, and after a year could no longer be obtained, though cetyl alcohol (melting-point 50° C.) could still be isolated. In the author's opinion the Storch reaction is, therefore, not conclusive of the purity of an oil when no coloration is obtained, although it should not be omitted in the qualitative examination of boiled linseed oil. C. A. M.

The Detection of Sesamé Oil in the Presence of Certain Dyestuffs. G. Fendler. (*Chem. Rev. Fettu. Harz-Ind.*, 1905, xii., 10, 11.)—Baudouin's test cannot be applied with certainty in the case of fats containing certain dyestuffs that give a red coloration with hydrochloric acid. In such cases the following modification of Soltsien's test (ANALYST, 1903, xxviii., 298) is recommended. The fat is treated with twice its volume of benzene and half its volume of the stannous chloride solution, and the test-tube placed in a bath at 40° C. until the tin solution separates, after which it is transferred to a bath at 80° C., where it is kept at such a depth that the level of the water outside is not above that of the stannous chloride solution inside. The heating is continued until the red coloration no longer increases in intensity. In the absence of sesamé oil the coloration caused by the dyestuffs soon disappears, and the stannous chloride solution remains colourless after further heating. C. A. M.

Differentiation of Cotton-seed Oil from Kapok and Baobab-seed Oils. E. Milliau. (*Ann. de Chim. anal.*, 1905, x., 9, 10.)—Kapok and baobab-seed oils reduce silver nitrate and give Halphen's reaction with greater intensity than even cotton-seed oil. A means of distinguishing them from the latter is based upon the fact that the fatty acids of kapok and baobab oils, after being washed and dried rapidly at 105° C., cause a strong reduction of a 1 per cent. alcoholic solution of silver nitrate even in the cold, whereas the fatty acids of cotton-seed oil do not effect appreciable reduction until heat has been applied. The fatty acids from kapok oil or baobab oil, mixed in the proportion of 1 per cent. with olive or other oils, give a dark-brown reduction after about twenty minutes, when 5 c.c. are thoroughly shaken with 5 c.c. of the solution of silver nitrate in absolute alcohol, and allowed to stand. On the other hand, the fatty acids from mixtures of olive oil with 10 to 50 per cent. of cotton-seed oil give no coloration under the same conditions. C. A. M.

Birch-leaf Oil. (*Haensel's Report;* through *Pharm. Journ.*, 1904, lxxiii., 900.) —Birch leaves yield 0·049 per cent. of an olive-green, optically inactive essential oil, containing 43 per cent. of a stearoptene. The oil, which liquefies at 35° C., and is soluble in warm alcohol, has a specific gravity of 0·9074 at 30° C., an ester number of 99, and a saponification number of 146·7. When freed from stearoptene, a thick oil of acid reaction remains of specific gravity 0·8732 at 20° C. A. R. T.

Tests for Wood Tar Pitch. B. M. Margosches. (*Chem. Rev. Fett u. Harz-Ind.*, 1905, xii., 5-9.)—The samples of pitch examined were derived from beech-wood tar, and had the following elementary composition: Bosnian pitch, C63·0 and H6·0 per cent.; and Hungarian pitch, C66·0 and H10·0 per cent. Wood tar pitch is thus richer in oxygen than other artificial "asphaltums." The behaviour of wood tar pitch with certain solvents affords a means of distinguishing it from the other artificial products. Thus, on treating 0·1 to 0·3 gramme with 10 to 15 c.c. of carbon bisulphide the liquid remains colourless for some minutes, and only acquires a faint yellow coloration after twenty-four hours at the ordinary temperature or at 30° C. Petroleum spirit is a still more characteristic reagent, for it remains colourless after standing in contact with the pitch for twenty-four hours, although it dissolves about 2 per cent. of the substance. Davies found that from 16 to 24 per cent. of wood tar pitch was soluble in that solvent, and the author attributes the discrepancy to the fact that more volatile products are now expelled during the distillation of the wood tar. Chloroform dissolves wood tar pitch in considerable quantity, yielding a dark brown solution. Alcohol and ether dissolves much less, and the solutions only turn yellow after some time. The Hungarian pitch only imparted a slight coloration to benzene, but the Bosnian pitch coloured the liquid yellow in five minutes, the colour changing to red after twelve hours. C. A. M.

The Differentiation of the Different Forms of Nitrogen in Glue. S. R. Trotman and J. E. Hackford. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1073.)—In addition to the determination of water and mineral matter, the authors consider that an analysis of glue should include determinations of the total nitrogen, nitrogen precipitated by zinc sulphate, and subsequently by bromine in the filtrate. Physical tests they consider to be in general unreliable. The authors proceed by analytical methods very similar to those which have been previously applied to the valuation of glue, etc., by A. H. Allen (*Comm. Org. Analysis*, vol. iv., 1898). A. R. T.

A Contribution to Rubber Analysis. W. Esch and A. Chwolles. (*Gummi. Zeitung*, 1904, xix., 125; through *Chem. Zeit. Rep.*, 1904, xxviii., 354.)—The author considers that the factor 100 : 97·5 in Henriques' formula is not invariably correct. This factor depends very largely on the solvent used for extraction, also on the nature of the caoutchouc in question and the method of vulcanization employed. Hot acetone is better than ether (Henriques) for the extraction of the unsaponifiable matter, as ether dissolves appreciable quantities of caoutchouc. H. A. T.

Mineral Constituents of Sumac. S. R. Trotman. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1137.)—Whilst it is of the greatest importance that sumacs should contain a due proportion of tannin, it is also desirable that a limit of ash should be fixed. The presence of sand and iron in sumac is actively deleterious, the former to the grinding machinery and the latter to the goods, which may become stained. Commercial sumacs, having an ash of 6 to 9·5 per cent., generally contain distinct amounts of iron removable by the magnet, sometimes 0·25 per cent. By moderate winnowing it is easy to obtain a sumac showing ash, 6·5 per cent.; silica, 0·75 per cent.; iron, 0·15 per cent. The winnowing process removes much silica, iron, etc. The combined iron in sumac is not more than 0·1 per cent. Adulteration of *sumac leaf with the stems does not cause any rise in the proportion of mineral* matter. A. R. T.

The Detection of Indoxyl (Indican) in Urine. G. Denigès. (*Bull Soc. Pharm.*, Bordeaux, September, 1904. *Ann. de Chim. anal.*, 1904, ix., 459, 460.)—Four c.c. of the urine are treated with basic lead acetate, then with 3 c.c. of hydrochloric acid (specific gravity, 1·17 to 1·18), 1 c.c. of chloroform, and a drop of a solution of potassium chlorate (5 grammes per litre). The tube is shaken, and after the addition of a second drop of the potassium chlorate solution, allowed to stand. If the chloroform is colourless or only slightly blue, indican is absent or only present in normal quantity. But if the chloroform has a pronounced blue colour, excess of indican is present. In such cases the liquid should be shaken with another drop of potassium chlorate and allowed to stand, and this treatment repeated until the colour of the chloroform does not increase in intensity after the addition of a fresh drop of the reagent. The coloration is proportional to the amount of indoxyl in the urine, the indoxyl being oxidized into "hemi-indigotin," which is readily soluble in chloroform, in which respect it differs from commercial indigotin. C. A. M.

Laboratory Methods. C. Goldschmidt. (*Chem. Zeit.*, 1904, xxviii., 1229.)—Tin dust, which is a more efficient reducing agent than many others, can be most conveniently prepared by allowing solutions of a tin salt to stand in aluminium vessels.

For the preparation of mono- and di-methylaniline the following method is recommended:

Formaldehyde acts on aniline, forming anhydro-formaldehyde-aniline, $C_5H_5NCH_2$, and this compound, by reduction with tin dust and hydrochloric acid, yields monomethylaniline. Treating this with formaldehyde and subsequent reduction, dimethylaniline is obtained. H. A. T.

INORGANIC ANALYSIS.

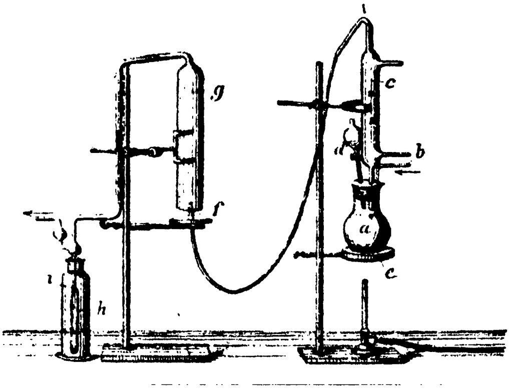
A Method for Detecting the Presence of Certain Metallic Oxides. C. R. C. Tichborne. (*Scient. Proc. Royal Dublin Soc.*, 1904, x., 331-334.)—The test proposed is based upon the fact that most metallic oxides react with a solution of sodium hydrogen carbonate, a certain quantity of sodium carbonate being formed. The presence of the latter may be ascertained by the addition of a little phenolphthalein. The solution of sodium hydrogen carbonate is prepared by dissolving 10 grammes of the pure salt in 100 c.c. of water. A few drops of a normal solution of nitric acid are added, as the solution alone will probably give a faint pink coloration with phenolphthalein owing to slight dissociation of the salt. The substance to be tested is triturated with a few cubic centimetres of this solution and filtered. If any of the oxides specified be present, the filtrate will be alkaline in reaction towards phenolphthalein. Red lead, mercurous oxide, cupric oxide, ferric oxide, manganese dioxide, and alumina give no reaction; whilst litharge, silver oxide, mercuric oxide (yellow and red), cuprous oxide, bismuth oxide, stannic oxide, antimony trioxide, ferrous oxide, manganous oxide, and zinc oxide readily decompose the sodium hydrogen carbonate. W. P. S.

The Determination of Tin, Antimony, and Arsenic in Ores and Alloys. Henri Angenot. (*Zeit. angew. Chem.*, 1904, xvii., 1274.)—Essentially the author's method consists in completely decomposing the ore or alloy by a single fusion with sodium peroxide, and separating tin and arsenic from antimony according to Rose's method. A. G. L.

The Detection of Ferrous Salts in the Presence of Ferric Salts. L. Blum. (*Zeit. anal. Chem.*, 1905, xliv., 10, 11.)—The well-known reaction for nitric acid cannot be applied conversely in the ordinary way for the detection of ferrous salts. For on adding sulphuric acid to a solution of ferrous sulphate, and then pouring nitric acid on to the surface, so much heat is produced as to prevent the formation of the brown colour. This is obviated by introducing a large crystal of potassium nitrate instead of the nitric acid. When a ferrous salt is present, red streaks changing to brown form round the periphery of the crystal, and the coloration does not disappear for several hours. C. A. M.

The Detection of Tin in the Stannous Condition. L. Blum. (*Zeit. anal. Chem.*, 1905, xliv., 10-12.)—The reaction for detecting ferrous salts (see preceding abstract) can be used indirectly for the detection of stannous salts. If a solution of, say, stannous chloride, containing hydrochloric acid, be treated with ferric chloride, a corresponding quantity of ferrous chloride is formed; and on adding to the solution an equal volume of concentrated sulphuric acid, cooling the mixture, and introducing a crystal of potassium nitrate, the characteristic red to brown coloration will be produced. C. A. M.

Estimation of Phosphorus in Calcium Carbide. Hj. Lidholm. (*Zeit. angew. Chem.*, 1904, xvii., 1452.)—The author found that in Gall's process a sudden evolution of CO_2 would cause the loss of part of the contents of the crucible; and when this was remedied by the employment of a large crucible he found it was not practicable to completely decompose the powdered carbide with the nitre. He therefore evolves the acetylene from the carbide with the aid of alcohol, which can be done sufficiently slowly to feed a burner with the gas. The following is the apparatus used: *a* is a flask of about 500 c.c. capacity, with a neck 50 millimetres wide, fitted with a three-holed stopper, which carries a tube for passing hydrogen (*b*), a reflux condenser (*c*), and a dropping funnel (*d*). The tube *b*



is somewhat bent, to enable a crucible (*e*) to stand on the bottom of the flask. The reflux condenser is connected by a tube (glass or indiarubber) with the burner *f*, the jet being so fine that the acetylene flame does not become smoky. Surrounding the burner is a glass cylinder (*g*) 32 centimetres long and 5 centimetres wide, which is continued above as a glass tube about 5 millimetres wide. This tube passes down into a wash-bottle (*i*), is there expanded into a long bulb (*h*), perforated with several apertures. The second tube from the wash-bottle is connected with a powerful filter-pump. Ten grammes of the powdered carbide are weighed into a crucible, which is then placed on the bottom of the flask, and the apparatus is put together, as shown in the diagram. Hydrogen is passed through the tube *b*, and when the air in the apparatus is expelled the hydrogen is lit, and at the same time the filter-pump is set in action. The condenser is then set in operation; from the funnel *d* about 30 c.c. of absolute alcohol are run in by drops, and afterwards about the same amount of water is cautiously dropped in. Any phosphuretted hydrogen and organic phosphorous compounds will be burnt to phosphoric acid, which will for the greater part be deposited on the walls of the cylinder. Any portion escaping from the cylinder will be completely retained in the wash-bottle.

After the evolution of gas in the flask is fully completed HCl is poured in to dissolve the precipitated lime, and the contents of the flask are brought to the boil, so that all the phosphorus compounds are carried to the burner. When the gas has escaped from the burner for about ten minutes the stream of hydrogen is discontinued, and glass cylinder, connecting-tube, and wash-bottle are washed with warm ammonia into a beaker. This solution is filtered, to remove the silica, and finally the phosphoric acid is precipitated in the usual way with magnesia mixture.

W. P.

Note on the Chemistry of High-percentage Ferrosilicon. Haas. (*Stahl. u. Eisen*, 1904, xxiv., 1315; through *Chem. Zeit. Rep.*, 1904, xxviii., 353.)—Fifty per cent. ferrosilicon is completely soluble in cupric-ammonium fluoride, with deposition of metallic copper; silicic acid does not appear to be separated. Eighty per cent. ferrosilicon is only slowly attacked. The effect of this solvent on carbon and phosphorus has not yet been examined.

H. A. T.

Note on the Amount of Moisture in Gases dried by Phosphorus Pentoxide. Edward W. Morley. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1171.)—The author showed in 1887 that the combined amount of moisture and phosphorus pentoxide vapour remaining in a gas dried by means of this reagent is about 1 milligramme per 40,000 litres. Since then, greater precision in working has been attained, and it was found that 4,300 litres of gas retained only 0·1 milligramme of water plus phosphorus pentoxide. In order to determine the water in this more accurately, 900 litres of hydrogen and oxygen were submitted to a very slow combustion (lasting a fortnight) in an apparatus designed by the author for the synthesis of water. As only 3 litres of the gaseous mixture entered the combustion chamber per hour, it was assumed that any volatilized phosphorus pentoxide would remain in contact with steam and water so long as to be completely absorbed. The ½-litre of water thus obtained was collected (with due precautions), and, after being made alkaline with a very little sodium carbonate, was evaporated to about 5 c.c. The molybdate precipitate from this quantity was found, by comparison with standards, to be equal to about 0·02 milligramme of P_2O_5. This proportion is practically identical with the sum of the moisture and phosphorus pentoxide vapour as determined some years ago. Although small errors may somewhat affect these results, yet no gravimetric experiments need take into account the moisture in a gas dried by this substance. A current of gas passing at the rate of 2 litres per hour through 25 grammes of phosphorus pentoxide, in a properly filled drying-tube, contains much less than 1 milligramme of moisture in 40,000 litres, but whether the actual amount is $\frac{1}{2}$ or $\frac{1}{10}$ milligramme cannot yet be stated. A. R. T.

APPARATUS.

Use of a Mercury Cathode in Electrolysis. Ralph E. Meyers. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1124.)—The author has extended the work of E. F. Smith (ANALYST, 1903, 372) to other elements. The beakers used in the electrolyses were of a form similar to those used by Smith, three sizes, holding 70, 45, and 35 cc., being employed. The procedure was substantially identical with that of Smith (*loc. cit.*), 70 to 80 grammes of mercury being used in each experiment.

By the use of a mercury cathode, chromium can be accurately determined in slightly acidulated (sulphuric acid) solutions. The liquid, measuring about 70 c.c., and containing about 4 drops of sulphuric acid, is electrolysed with a current of 0·4 ampère and 7·5 volts. The amount of chromium present should not greatly exceed 0·2 gramme. As the chromium amalgam readily decomposes water, it should be washed rapidly, and the alcohol used subsequently for this purpose should be fairly anhydrous. Chromium may by this method be separated from large or small amounts of aluminium, which latter may then be precipitated from the solution after electrolysis in the usual way. The strength of the current during the last hour or two should be increased (0·4 ampère to 0·8 ampère). Similarly, beryllium remains in solution, and the chromium may be deposited when in admixture with it. Iron may also be separated from beryllium in the same way.

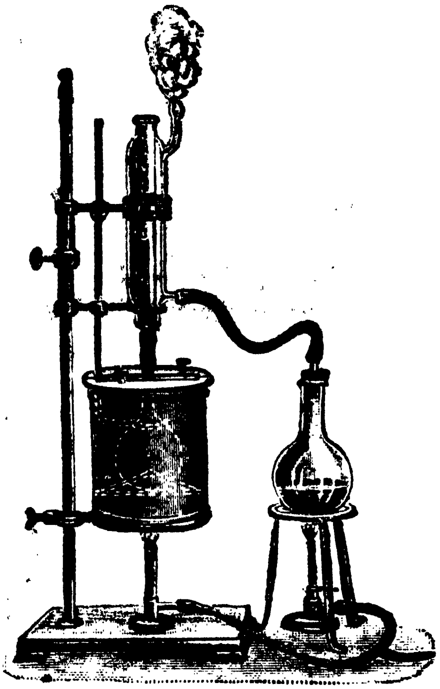
Molybdenum forms a brilliant white amalgam with mercury, and can be deposited from a sulphuric acid solution of a molybdate, or from the acidified aqueous solution of molybdenum trioxide. Overnight deposition (fourteen hours) is usually resorted to, using a current of not more than about 1·5 to 2·0 ampères and 6 volts. A slight solvent action of the acid on the mercury, due to the heat evolved, is noticed if a stronger current be employed. About 5 drops of sulphuric acid for each 10 c.c. of liquid are added. It is best to neutralize some of the acid towards the end, since too great acidity retards the deposition of the molybdenum. Vanadium may be separated from molybdenum by the above method. Vanadium solutions turn blue, but no deposit occurs under these conditions. Thus the pure blue colour of the liquid shows when all the molybdenum has been deposited. Iron may be separated from vanadium by this method.

Similarly, it is found that iron may be accurately separated from cerium, lanthanum, praseodymium, neodymium, and yttrium. The current employed is

0·8 ampère and 6 volts, with 8 drops of sulphuric acid in the liquid. About fourteen *hours, or less, according to the iron present, is necessary for the electrolysis.*

The value of Drown and McKenna's separation of iron and aluminina (*Journ. anal. Chem.*, v., 627) was fully confirmed by the author. The test analyses on all the above methods of separation were very satisfactory. A. R. T.

New Apparatus for the Acoustic Determination of Vapour Density. R. Wachsmuth. (*Chem. Zeit.*, 1904, xxviii., 869.)—The apparatus consists of a steam-jacketed cylinder, or tube, in the lower end of which a standard metal whistle or reed is fixed. The whistle is connected by a rubber stopper or piece of tubing to the vapourising flask, and the whole can be lowered into a closed water-bath.



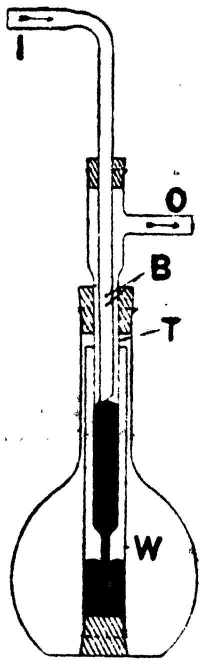
For the determination of the vapour density of a liquid at 100° C., 10 to 20 c.c. of the sample are brought into the vapouriser; this is connected to the jacketed whistle, and when this and the bath are at 100° the whole is lowered into the water-bath. At once, or very shortly (according to the boiling-point of the substance), the internal pressure causes the whistle to sound, the note depending on the relative pressure of the vapour in the flask and that of the air. The note obtained is compared to that of a standard adjustable whistle, and the vapour density thus directly determined. The density of the vapour is to that of air practically inversely as the square of the rate of vibration observed.

The author gives exact formulæ for the calculation of results, and describes the process in considerable detail.

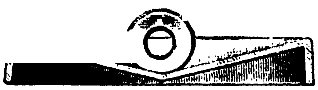
The apparatus is obtainable from Messrs. Max Kaehler and Martini, Berlin, W. H. A. T.

An Electrical Heater for Laboratories. S. R. Trotman and J. E. Hackford. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1137.)—This simple contrivance consists of a tin about 6 or 8 inches deep, of sufficient diameter to hold a lamp of 16, 32, or 50 candle-power. The exterior of the tin is covered with asbestos-paper. Ordinary type lamps are employed, but those which have become discoloured by long usage give the most heat, and these can be obtained at a small cost. The lamp is fixed through a hole near the bottom of the tin, and the whole apparatus is placed on a wooden base, the top surface of which, next to the tin, is covered with asbestos. The lamp is useful for saponifications, fat-extractions, alcohol and ether distillations, etc., the source of heat being quite constant and unaffected by draughts, while no ignition of inflammable vapours can take place. A. R. T.

A Simple Regulator for Use with Large Gas Burners. William H. Sodeau. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1136.)—As shown in the figure, the regulator consists of a 16-ounce Bohemian glass flask, inside of which a glass tube (W) is placed, open at the top and closed at its lower end by a cork which rests on the base of the flask. The neck of the flask is closed by a rubber stopper carrying a tube (T) of the shape shown in the figure. The narrow (bottom) end of this tube reaches nearly to the cork in W. The middle portion of the tube T has a diameter of 11 millimetres, being 3 millimetres greater than the external diameter of I. The upper and wider part of T has a side tube (O), at which the connection with the burner is made. The top of T is closed by a cork carrying the tube I, through which the gas supply comes, the end of the tube being cut off obliquely. Two small holes (B), of 0·8 and 1·2 millimetres diameter, allow the passage of sufficient gas to keep the burner lit in case the main gas supply is temporarily cut off by the regulator. Either of these holes may be blocked with soap if desired. To fit the regulator for use, the stopper is removed from the neck of the flask, which is then completely filled with methylated spirit heated to a temperature slightly higher than the regulator is likely to attain while in use. The stopper is then replaced, and sufficient mercury poured through the top of the tube T, as shown in the figure. The interior of the tube is dried by means of filter-paper. The regulator is next cooled to the temperature it is required to maintain, and the cork carrying the tube I placed in position in such a way that the end of the tube just touches the surface of the mercury. If the temperature increases 1° C., the expansion of the spirit causes the mercury in T to rise 5 millimetres, sufficient to make a great difference in the amount of gas passing. It is desirable in the first adjustment to hang a small thermometer in the flask to accurately register the temperature of the spirit. If the temperature falls 1° C. too low, the tube I is raised about 5 millimetres, and proportionately in other cases. The size of the regulator, which is very sensitive, may be largely varied according to requirements. A. R. T.



An Improved Form of Crucible Lid. The lid is made slightly convex to the crucible, so that any liquid splashing on the lid, during effervescence or evaporation of the contents of the crucible, drops back into the latter from the centre of the lid. The form of the lid does not in any way interfere with its general usefulness. It may be obtained from Messrs. J. J. Griffin and Sons, Limited, Sardinia Street, W.C. W. P. S.

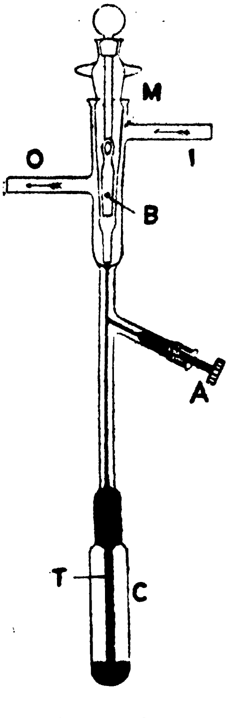


Material and Form of Rotating Cathodes. H. E. Medway. (*Zeits. anorg. Chem.*, xlii., 110.)—The author has tried to use silver and nickel crucibles as rotating cathodes instead of platinum crucibles (*vide Amer. Journ. Sci.*, xv., 320). With silver good results were obtained in electrolysing copper sulphate solutions ;

and on removing the copper by scraping it off as far as possible, and then treating with boiling hydrochloric acid, the loss in weight of the crucible was only 2 or 3 milligrammes. With nickel, on the other hand, the results were not good, the error being about 1 per cent.; and on removing the copper with nitric acid, the loss in weight of the crucible varied from 6 to 32 milligrammes.

Using rotating platinum *discs* as cathode, and a platinum wire bent around the edge of the disc in the form of a semicircle as anode (Shepherd, *Journ. Phys. Chem.*, vii., 508), the author found that the deposits of copper obtained easily fell off the disc owing to unequal deposition. With an anode of foil under and parallel to the disc, on the other hand, good deposits were obtained, but the author prefers the use of a crucible. It was found that aluminium discs (Hough, *Journ. Amer. Chem. Soc.*, xx., 302) could not be used. A. G. L.

A Highly Sensitive Modification of Novy's Gas Regulator. William H. Sodeau. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1136.)—The ordinary forms of mercurial gas regulators are ill-adapted to requirements when marked fluctuations in the gas pressure take place, but by employing one with a larger bulb, containing a liquid of high expansibility, the defects are overcome.



The modification shown in the figure has a cylindrical bulb (C) of about 15 to 20 c.c. capacity. (Any old-pattern regulator may be improved by cutting off the bulb and sealing on the lower portion of a Tollens gas regulator.) The inner tube (T) reaches nearly to the bottom of the bulb, and is continuous with the stem above. The bulb contains toluene, or a hydrocarbon or alcohol of higher boiling-point, and a little mercury. The bore of the lower part of the stem should be of such width as to hold about 2 to 3 c.c. of mercury, so that no gas is drawn into the bulb when the regulator cools. The side-tube holding the screw-adjustment (A) should be inclined at an angle of 20° to 30° from the horizontal, to retain mercury in the tube after cooling, otherwise a bubble of gas may be entrapped when the regulator is used again. The other details of the regulator remain unaltered. Since toluene has a coefficient of expansion about five times as great as that of mercury, and the capacity of the bulb has been increased ten times, this pattern of regulator will be about fifty times more sensitive than the ordinary form. If it is necessary to keep the bulb of the same diameter as the stem, still the substitution of toluene for mercury will give an instrument five times as sensitive. This regulator is very satisfactory for laboratory use, and its efficiency is enhanced if a pressure regulator is attached to the gas-supply pipe. A. R. T.

A Further Investigation of the Rotating Cathode. H. E. Medway. (*Zeits. anorg. Chem.*, xlii., 114.)—The author has applied the method previously described (*Amer. Journ. Sci.*, xv., 320) to other metals. A platinum crucible dipping into the liquid and rotating at a speed of 650 to 700 revolutions per minute was used as cathode. For *cadmium*, 0·2 gramme of cadmium sulphate was dissolved in 50 c.c. of water, and 10 drops of dilute sulphuric acid were added. A current of $C.D._{100} = 5$ and 6·6 ampères was used for fifteen minutes. Before stopping the current, the solution was made slightly alkaline with ammonia to avoid redissolution of the metal. For *tin*, 20 c.c. of a solution of stannous ammonium chloride, containing 0·08 or 0·16 gramme of tin, were mixed with 100 c.c. of a saturated solution of ammonium oxalate, and electrolysed with a $C.D._{100}$ of 6·6 to 11·6 ampères for twenty or fifteen minutes. In the case of *zinc*, 0·1 gramme of the metal as sulphate and 4 grammes of potassium oxalate were dissolved in 50 c.c. of water, and electrolysed with a current of 6·6 to 8·3 ampères for twenty-five or thirty minutes. The presence of ammonium salts had a retarding influence on the deposition. It was found that the zinc could be directly deposited on platinum, and removed without injuring the latter. For *gold*, 25 c.c. of gold chloride, containing 0·07 gramme of gold, were treated with a large excess of potassium cyanide and 30 drops of strong ammonia and electrolysed, using a current of 1·8 to 6·6 ampères for thirty minutes. In all cases the results obtained were excellent. Five gold determinations showed a maximum error of 0·0001 gramme, with an average of ±0·00005 gramme.

A. G. L.

Vitro-Ink.—A non-corrosive ink for writing on glass, celluloid, etc., which will be found very handy for many laboratory purposes. After being properly applied it is insoluble in water, strong acids, and only very slowly by strong alkalies. We find that in order to obtain the best results it is necessary to rigidly adhere to the directions supplied with the ink. The makers are Messrs. J. Griffin and Sons.

W. J. S.

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HIGH COURT OF JUSTICE.

KING'S BENCH DIVISION.

(*From the "Times" of Saturday, February* 4, 1905.)

(Before the LORD CHIEF JUSTICE OF ENGLAND, MR. JUSTICE KENNEDY, and MR. JUSTICE RIDLEY.)

BAYLEY *v.* COOK.

THIS was a case stated by the Justices of Andover on an information laid by the appellant Bayley, an inspector under the Food and Drugs Acts, against the respondent Cook, for wilfully selling to the appellant, to his prejudice, milk not of the nature, substance, and quality demanded, being deficient in milk-fat to the extent of 53 per cent. It was objected on behalf of the respondent that the analyst's certificate was defective, owing to its not stating the standard upon which it was based. The material parts of the certificate were as follows: "I am of opinion that the sample contained the parts as under: Milk-fat, 1·4 per cent.; milk solids other than milk-fat, 5·6 per cent. *Observations.*—This milk is deficient in milk solids other than milk-fat to the extent of 2·9 per cent., which is equivalent to the addition of 34·2 per cent.

of water. It is also deficient in milk-fat to the extent of 53·4 per cent of the milk-fat." The justices dismissed the summons on the ground that the certificate was bad.

Mr. C. A. Russell, K.C. (Mr. W. J. H. Brodrick with him), argued that the certificate was sufficient. Under Section 4 of the Sale of Food and Drugs Act, 1899, the Board of Agriculture had power to make regulations for determining what deficiency in any of the normal constituents of genuine milk should raise a presumption that it was not genuine. Rule 1 of the Sale of Milk Regulations, 1901, made under that section, was as follows: "When a sample of milk (not being milk sold as skimmed or separated or condensed milk) contains less than 3 per cent. of milk-fat, it shall be presumed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1899, until the contrary is proved, that the milk is not genuine, by reason of the abstraction therefrom of milk-fat or the addition thereto of water." The certificate, taken in conjunction with this rule, was *primâ facie* evidence that an offence had been committed. He cited "Fortune *v.* Hanson" (1896, 1 Q.B., 202).

The respondent did not appear.

The Lord Chief Justice, in giving judgment on Wednesday, said that he did not wish it to be thought that the provisions of the Act of 1899 in any way lessened the obligation upon the analyst as to what he was bound to state in the certificate by Section 18 of the Sale of Food and Drugs Act, 1875. If nothing more appeared than "milk-fat, 1·4 per cent.," he doubted whether that would be a sufficient certificate. It was necessary to show that the sample had been compared with some standard, and he gathered that that was what misled the justices, for they seemed to have thought that the basis on which the certificate was framed was not sufficient. It would have been more correct to state in the certificate what the proper quantity of milk-fat was, but, as a little examination would show, it did appear from the certificate, and it did correctly show how much milk-fat was deficient, and the standard of calculation could be gathered from the certificate itself, and therefore the objection to it was not a good one.

Mr. Justice Kennedy, in agreeing, said that it was important that the accused should know without elaborate calculations what he was accused of, and he hoped that those who gave certificates would err, if at all, on the side of fulness and simplicity.

Mr. Justice Ridley agreed.

The appeal was accordingly allowed and the case remitted to the magistrates.

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APRIL, 1905. Vol. XXX., No. 349.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual meeting of the Society was held on Wednesday evening, March 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. Thomas Fairley, occupied the chair.

Messrs. J. H. B. Jenkins and Arthur Tighe were appointed scrutators of the ballot-papers for the election of Officers and Council.

The HON. TREASURER (Mr. E. W. Voelcker, A.R.S.M.) presented his annual report, with the accounts of the Society for the year 1904.

On the motion of Mr. DIBDIN, seconded by Mr. BLOUNT, the Hon. Treasurer's report was unanimously adopted, and a vote of thanks accorded to the Hon. Treasurer for his services during the year.

The HON. TREASURER, having acknowledged the vote of thanks, proposed a vote of thanks to the auditors, Mr. W. P. Skertchly and Mr. C. G. Moor, M.A.

Mr. RICHARDS seconded this, and the vote was unanimously passed.

On the motion of Dr. VOELCKER, a vote of thanks was unanimously passed to the Hon. Secretaries for their services during the year.

Mr. BEVAN acknowledged the vote of thanks.

The PRESIDENT moved, and it was unanimously agreed, that a vote of thanks be accorded to the President and Council of the Chemical Society for their kindness in allowing the Society the use of their rooms at Burlington House during the past year.

The PRESIDENT delivered his annual Address.

Dr. DYER proposed that a very hearty vote of thanks be accorded to the President for his Address, and that his permission be requested to publish the Address in the ANALYST.

Mr. FISHER seconded the resolution, which was carried unanimously.

The PRESIDENT briefly responded.

The scrutators having reported to the President the result of their examination of the ballot-papers,

The PRESIDENT announced that the Officers and Council for the ensuing year had been elected as follows:

President.—E. J. Bevan.

Past-Presidents (limited by the Society's Constitution to ten in number).—M. A. Adams, F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Thomas Fairley; W. W. Fisher, M.A.; Otto Hehner; Alfred Hill, M.D.; J. Muter, Ph.D.; Sir Thomas Stevenson, M.D., F.R.C.P.; J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Vice-Presidents.—L. Archbutt, B. Kitto, W. J. Dibdin.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—Alfred C. Chapman and P. A. Ellis Richards.

Other Members of Council.—Julian L. Baker; R. Bodmer; R. Hellon, Ph.D., A.R.S.M.; G. T. Holloway, A.R.C.S.; J. H. B. Jenkins; E. W. T. Jones; S. Rideal, D.Sc.; Alfred Smetham; J. E. Stead, F.R.S.; R. T. Thomson; L. T. Thorne, Ph.D.; John White.

Mr. Fairley then vacated the chair in favour of the newly-elected President, Mr. Bevan.

Mr. Bevan expressed his acknowledgment, and the proceedings then terminated.

The monthly ordinary meeting of the Society was held on Wednesday evening, March 1, in the Chemical Society's Rooms, Burlington House, immediately after the annual meeting. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. E. Hinks and S. E. Melling were read for the second time; and certificates in favour of the following candidates were read for the first time: Edward Richards Bolton, 16, Flanchfort Road, Ravenscourt Park, W., chief chemist to the East Indian Products Co., Ltd., at Albert Mills, Hammersmith; John Thomas Dunn, D.Sc. (Dunelm), F.I.C., 10, Dean Street, Newcastle-on-Tyne, joint Public Analyst for the Cities of Durham and Newcastle-on-Tyne, and for the Boroughs of Gateshead, Sunderland, South Shields, Berwick-on-Tweed, and Tynemouth; Edward Kenneth Hanson, M.A. (Cantab.), A.I.C., Mapledene, Hadley Wood, Barnet, Analyst and Lecturer on Physical Chemistry at University College, Reading; Frederick Thomas Harry, 106, Sandmere Road, Clapham, S.W., assistant to Mr. Alfred C. Chapman; the Hon. Francis Robert Henley, M.A. (Oxon.), A.I.C., 6, Melville Street, Edinburgh, chemist at Messrs. Jeffrey's Brewery, Roxburn, Edinburgh.

The following papers were read: "The Estimation of Oxygen in Copper," by S. Dickson; and "Some Conditions affecting the Ether Value of Brandies," by Philip Schidrowitz, Ph.D., and Frederick Kaye, A.R.C.Sc.

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ANNUAL ADDRESS OF THE RETIRING PRESIDENT.

Delivered at the Annual Meeting, February 1, 1905.

Last year I was in the fortunate position of being able to say that we had not lost any members by death during my term of office. Now at its close I regret to have to report the death of six ordinary members and one honorary member, Dr. A. W. Williamson. During the year we have elected fourteen new ordinary members, and our numbers now stand at:

Honorary members	8
Ordinary members	301

Our loss by death is the greater as it includes Mr. Allen and Mr. Chattaway, both members of the publication committee. Mr. Allen was one of the founders of our Society, and has held its chief official positions. That he was one of the most active workers in the ranks of our profession is well shown in the able and sympathetic review of his work by Mr. Hehner, a tribute more eloquent of Mr. Allen's untiring energy than anything that I can say. It is indeed difficult to overestimate how much practising analysts throughout the world owe to him for his work in testing and collating processes and methods of organic analysis.

Mr. W. Chattaway, who has passed away in middle life, was at one time associated with Mr. Allen, and was an active worker in several directions. He held qualifications as a pharmaceutical chemist, and had special knowledge in the chemistry of drugs. In recent years he occupied the position as Head of the Laboratory of the Apothecaries' Hall. His amiable qualities endeared him to his friends, and his services will be much missed in many directions. He held the appointments of Public Analyst for the Boroughs of Hammersmith and Colchester.

Dr. A. P. Aitken, an old member of our Society, was Chemist to the Highland and Agricultural Society of Scotland, Professor of Chemistry at the Royal (Dick) Veterinary College, Edinburgh, and Public Analyst for several of the counties in Scotland. He received his training at the Universities of Edinburgh and Heidelburgh, and was the author of numerous papers published in the *Highland and Agricultural Society's Journal*, and occasionally in the ANALYST.

Mr. T. A. Pooley was educated first in France, and afterwards at King's and University Colleges, London. For a time he edited one of the brewing journals, and afterwards frequently contributed papers to them. He held the positions of Public Analyst for the County of Essex and the Borough of West Ham, and has contributed papers at various times to the ANALYST.

Mr. H. S. Carpenter formerly practised as an analyst in London, and contributed a number of papers to the earlier volumes of the Journal. For some years before his death he had retired from the ranks of our profession.

Mr. Edgar B. Kenrick held the position of Professor at St. John's College, Winnipeg, Canada. His contributions to the ANALYST related to the detection of cocoanut oil and of cotton-seed oil in lard.

The financial position of our Society is approximately the same as at this time last year. Owing to a number of special reasons our expenses have been heavier in each department, as shown in the statement carefully compiled by the Hon. Treasurer, to whom our best thanks are due. It is our duty to try to improve the position of our Society, and to increase the value of its Journal. Thus, we have spent £31 more on the ANALYST in 1904 than in 1903. Our ideal is to make this paper the best of its kind in the world, and an increased expenditure should be regarded as well-spent money, even if we only attain partial success in this direction. It has been decided at the close of the present year to increase the price of the Journal to non-members, and due notice of this change will be given to subscribers.

Your Council have carefully considered the matter of the Incorporation of the Society under the Limited Liability Companies' Act, and they have taken the necessary steps towards preparing the documents required for registration under that Act. The draft scheme will, it is hoped, be shortly submitted to the members, and considered at a special meeting called in accordance with the rules of our Society and the requirements of the Act. The Journal and funds belonging to the Society are now held for us by trustees. We are grateful to, and in no way dissatisfied with, those who have taken this responsibility, but it is felt that the position and aims of the Society can be better secured and maintained by incorporation under the clause permitting "associations not for profit" to register without using the adjective "limited."

The following papers have been presented to the Society during the year:

"Note on Chinese Tallow-seed Oil." By L. Myddleton Nash.
"Note on the Estimation of Mechanical Wood-pulp in Paper." By C. F. Cross and E. J. Bevan.
"Note on the Analysis of Jam." By Raymond Ross.
"The Microscopic Examination of American Cotton-seed Cake." By A. L. Winton.
"The Composition of Milk." By H. Droop Richmond.
"The Estimation of Morphine in Opium." By Philip Schidrowitz, Ph.D.
"The Iodine Absorption as a Factor in the Examination of Otto of Roses." By F. Hudson-Cox and W. H. Simmons.
"Analyses of Potable Spirits used by the Native Population in India." By Harold H. Mann.
"A Rapid Accurate Method for the Volumetric Estimation of Carbon Dioxide." By Thomas Macara.
"The Microscopic Examination of Metals," illustrated by Lantern Slides. By J. H. B. Jenkins and D. G. Riddick.
"Cod-liver Oil and other Fish Oils." By J. F. Liverseege.
"Note on Mushroom Ketchup." By J. F. Liverseege.
"Note on some Constants obtained in the Examination of Margarine." By Edward Russell, B.Sc., and V. H. Kirkham, B.Sc.
"Note on the Estimation of Sugars in Concentrated Malt Extract." By Arthur R. Ling and Theodore Rendle.
"A 'Beer-Beater' for Use in Original Gravity Determinations." By John S. Ford, F.R.S.E.
"Roasted Beetroot." By E. G. Clayton.
"The Analysis of Sweetened Condensed Milk." By J. B. P. Harrison.
"A Collection of Readings with the Zeiss Oleo-Butyrometer." By the late W. Chattaway and C. G. Moor, M.A.
"Note on the Estimation of Sugars and Starch in Vegetable Substances." By John S. Ford.
"Improved Continuous-Observation Polarimeter Tube." By John S. Ford.
"Further Note on Mushroom Ketchup." By J. F. Liverseege.
"The Detection and Estimation of Small Quantities of Maltose in the Presence of Dextrose." By Julian L. Baker and W. D. Dick.
"The Use of Palladium-Hydrogen as a Reducing Agent in Quantitative Analysis." By Alfred C. Chapman.
"Some Abnormal Milk Results." By Sidney Harvey.

"Electric Furnaces for Laboratory Use." By Bertram Blount.

"Note on Commercial Amyl Alcohol." By H. Droop Richmond and J. A. Goodson.

The following exhibits were shown at the meetings:

"An Improved Form of Tin for the Collection of Samples under the Sale of Food and Drugs Act." By the late W. Chattaway.

"A Double Surface Condenser." By Herbert E. Burgess.

The following reprints were inserted in the Journal:

"The Determination of Boric Acid in Cider, Fruits, etc." By the late Alfred H. Allen and Arnold R. Tankard (read at the British Pharmaceutical Conference, 1904).

"Results of Experiments on the Effect of Boric Acid and Borax administered with Food" (from Circular No. 15, issued by the United States Department of Agriculture).

Judgment of the High Court of Justice, King's Bench Division, in the case *Hull* v. *Horsnell*, relating to Copper in Preserved Peas (from the *Times* of November 7, 1904).

The list of papers for the year is good, but their number, I regret to say, has remained somewhat stationary for several years past. We are still much below the proportion of one paper from each member every five years. I know well the difficulty there is to find time in a busy laboratory for many kinds of original work, and I willingly admit my own shortcomings in this respect. At the same time, I earnestly press each member to do his best to increase his contributions to the Journal. In contributing a paper giving an account of honest work an author may benefit his fellows, but he benefits himself most of all. In this matter the splendid example set by Mr. Allen is surely one we should all try to follow, even if we must, many of us, come far short of his generous limit. The best way of showing our gratitude and appreciation of the work of those who have gone before us is to do something to add to what they have done for their fellow-members.

The paper by Messrs. Jenkins and Riddick on "The Microscopic Examination of Metals" is one which we are proud to have had read before our Society, and, with its beautiful illustrations, forms a fitting commencement to what we hope may prove to be an enhanced and improved volume of the Journal. Another paper of great scientific interest was given by our hard-working Secretary, Mr. Chapman, on "The Use of Palladium-Hydrogen in Analysis." This and the paper by Messrs. Baker and Dick relating to the estimation of sugars are the first-fruits of the investigation scheme initiated by the Council, and form a most excellent beginning. I am happy to say the scheme has been taken up very kindly by the professors and teachers at the leading colleges in London, and we may hope for a series of useful papers or notes on analytical problems and methods of analysis, the work to be carried on under the supervision of the professor by selected students.

During the past year more than half the papers presented have been related to the analysis of food and drugs.

In addition to these original papers and reprints, abstracts of the following papers have been given in the Journal:

Food and drugs analysis	127
Toxicological and bacteriological analysis	6
Organic analysis	112
Inorganic analysis	156
Apparatus	31
Total number of abstracts	432

I am sure every member and every reader of the Journal will join with me in testifying our thanks to the Publication Committee and our gratitude to the Editor, for their labours during the year, not forgetting the most useful work done by the abstractors.

The question of the purity of the food and drink of the people continues to attract more and more public attention—a welcome change from the apathy with which such matters were formerly regarded. We still wait, however, for the enforcement of the recommendations of the Committee on the Use of Preservatives in Food. Recently the United States Department of Agriculture has published the first part of an elaborate report on "The Influence of Food Preservatives and Artificial Colours on Digestion and Health," drawn up by Dr. Wiley, the Chief of the Bureau of Chemistry. An abridgment of this report has already appeared in the ANALYST, but I may here repeat the final sentence of the report:

"It appears, therefore, that both boric acid and borax, when continuously administered in small doses for a long period, or when given in large quantities for a short period, create disturbance of appetite, of digestion, and of health."

If this be the case as regards boric acid and borax, possibly among the least detrimental of these preservatives, what shall we say of salicylic acids and salicylates, already forbidden by some of the Continental nations in articles for home consumption in their own countries? The processes of digestion require certain changes to take place in the food easily and without resistance, whereas the function of preservatives is to resist all such changes. In view of the uncertainty now prevailing, some of our local authorities have—I think not without reason—decided that no prosecutions shall be instituted in regard to preserves and similar foods where sugar is saved by the addition of small quantities of salicylic acid. We may hope that Dr. Wiley and his department may continue their valuable investigations, and that some day our authorities may decide that the health of our people is much more important than any extension of food manufacture founded on the use of preservatives.

One of the chief recommendations of the Select Committee, which sat in 1896, on Food Products Adulteration was that a Board or Court of Reference, preferably a standing Departmental Committee, be constituted, with advisory powers in matters relating to standards and analysis of food and drugs. This recommendation was further strongly supported by the Royal Commission on Arsenical Poisoning which sat 1901-1903, and the proposal has throughout been advocated by the Council of this Society. With the exception of the limits for milk and butter issued by the Board of Agriculture, the following words are still as true as when they were given out by your Council in 1897: "At the present time each individual Public Analyst is left to decide what does or does not constitute adulteration, what is or is not to be regarded as injurious to health, and what limits and standards are to be adopted in judging of the genuineness or otherwise of samples submitted to him. Disputed questions of the greatest scientific intricacy are now fought out, more or less imperfectly, in open court at the expense of vendors or public authorities, and, as a consequence, questions which might have been settled, to the benefit of the community, many years ago are still undecided, after more than twenty years' working of the Food and Drugs Acts."

Quite recently an example has arisen showing the uncertainty which exists. One of the recommendations agreed to by the Select Committee in 1896 was that the British Pharmacopœia should "be legally constituted the standard for drugs, and that deviations from such standard should be permitted only upon the production of a prescription of a duly qualified medical practitioner." It is true that this recommendation was not incorporated in the Act of 1899, but, considering the authorities responsible for the Pharmacopœia, analysts have, I think, rightly used it as a standard. A sample of cod-liver oil was condemned by a Public Analyst as not conforming to the Pharmacopœia tests. On reference to the Government Laboratory, the chemists there stated: "The sample does not conform to the tests specified in the British Pharmacopœia, 1898, for the examination of cod-liver oil." On further examination and analysis by the best authorities on this subject, it was given in evidence that the British Pharmacopœia tests were worthless as tests for the purity of cod-liver oil, and were devised at a time when the oil was made from putrid livers, and had been copied into successive editions of the Pharmacopœia. Does not this case prove the urgent necessity for a properly constituted authority to supervise all such matters?

In the Transactions of the Highland and Agricultural Society of Scotland for 1904 are three papers bearing on the milk question:

"The Influence of Food on Milk." By Allan Barnes Graham, junior.

"Milk Records." By John Speir.

"Milk Investigations at Garforth, 1903." By Charles Crowther, M.A., Ph.D.

In the first of these papers the author pleads for the upholding of the milk standards introduced by the Board of Agriculture, in the interests of the dairy industry itself. He describes a large number of experiments with individual cows fed alternately with poor and with rich rations.

The average percentages of fat in the milk were:

With Poor Rations.			With Rich Rations.		
1	2	3	1	2	3
2·92	2·95	3·02	3·36	3·37	3·20

Except at pasture, the quantity of water given to the cows is noted. The writer's view is that it is mainly by regulating the quantity of water consumed by the cow that we can alter the quality of the milk. To get a large yield of rich milk, use food rich in oil and albuminoids. The milk varies according as the cows are fed—in fact, the milk can be watered through the mouths of the cows.

Mr. Speir's paper on milk records gives the results of constant testing of the milk of 1,342 cows, chiefly of the Ayrshire breed, located on farms in Ayrshire, Dumfriesshire, and Wigtonshire. Full details are given of the effect of age and varying conditions on the milk yield. The records extended over periods of three months and six months, and the proportion of cows who ever gave morning milk under the 3 per cent. standard was less than 1 per cent. The average of all the cows for twenty-six weeks was 3·65 per cent. of fat in the milk.

These two papers can, I think, be well set against the low results chronicled from the farm at Garforth. I venture to suggest that in the latter there is something requiring explanation; it may be in some detail of the housing, feeding, or milking, or in the health or selection of the cattle at this experimental farm. Other milks in the same district do not show the same low results. The consumption of milk in the city of Leeds is very great, and its centre is seven miles from Garforth. The following are the averages for the past two years of testing many hundreds of samples of milk, including in the average the adulterated samples:

	1903.	1904.
Average percentage of fat in the milk ...	3·53	3·68
Percentage of adulterated samples	20·3	23·8

These adulterated samples were not only lower than the limits specified by the Board of Agriculture, but in each case the ash and the nitrogen of the milk were carefully determined and were found deficient.

The report of the Departmental Committee appointed a year ago to inquire into the Working in Great Britain of the Fertilizers and Feeding Stuffs Act, 1893, has now been issued.

The Committee recommend that powers be given to local authorities to use public funds for the purposes of the Act, to appoint official samplers to take samples on behalf of purchasers, or to take test samples on behalf of the local authority. The procedure recommended is similar to that under the Sale of Food and Drugs Act, but where articles differ from the guarantee, copies of the certificates are to be sent to the Board of Agriculture, whose approval is necessary before instituting a prosecution. Purchasers, or associations of purchasers, may submit samples for analysis, taken with or without the formalities required under the Act, but in the latter case the analyst shall have power to charge an enhanced fee. Sellers are required to state "the actual constituents present in fertilizers and feeding stuffs." This, I presume, means the actual proportions or percentages of constituents. If so, one exception is made, I think, without sufficient reason, in regard to the oil and albuminoids present in foods consisting of a particular seed, either whole or ground, but not otherwise altered.

That the Board of Agriculture take steps to secure uniformity of procedure in the analysis of fertilizers and feeding stuffs, and issue a statement of limits for each constituent of the principal fertilizers and feeding stuffs.

The district agricultural analysts must specially endorse their certificates when there is such a difference between the analysis and the guarantee as either to render inquiry desirable or to give a *primâ facie* case for a prosecution.

Consistently with securing the services of competent men, the analyst is to reside in the district for which he acts, so as to afford opportunity for farmers and others to consult him. In my experience as analyst to an agricultural society before this Act came in force, and as an analyst under the Act, there are very few personal consultations ever required, and advice is more frequently given by letter than personally. Unless in the future the amount of work arising under the Act is increased beyond any comparison with what it has been up to the present time, I think it will be difficult to find competent men to devote themselves entirely to work under this Act, and, as a rule, the men who now hold these appointments locally derive their chief income from other sources.

In determining the value of basic slag, the proportion soluble in 2 per cent. citric acid is to be noted.

In regard to agricultural analyses, I have found farmers sometimes averse to stating the price at which they bought the articles. With samples taken under the Act the price must be stated, and I think this is most desirable in all cases, whether samples be taken under the Act or not. I have met with some extraordinary cases where the analysis and guarantee were quite in agreement, but the price charged was twice or three times the market value. The vendor in these cases traded on the ignorance of his customers, and simply laughed at the threat of proceedings being taken.

It is, I think, very evident that the Board of Agriculture through its staff are most anxious to do all they can in the interests of the farmers and as regards farming produce of all the citizens of the country. It might tend to promote this end if copies of every publication issued by the Board were sent to each agricultural or public analyst, even when these publications do not bear directly on matters relating to analysis. Many of us have to acknowledge the courtesy of the Government Departments in sometimes sending us copies of reports relating to the work in which we are engaged. This society is really a co-worker with these departments, and any trouble or expense incurred in a more liberal circulation of reports or papers would in due time be well repaid.

The Supplement to the Thirty-Second Annual Report of the Local Government Board, drawn up by Dr. Power, the medical officer of the Board, contains reports of a number of investigations of great interest to those engaged in the sanitary examination of air and water.

In Appendix A, No. 11, report by Dr. Bulstrode, "On the Water-Supply of Bridgend, Glamorganshire, and the Relation thereto of the Prevalence of Enteric Fever."

Report by Dr. Gordon, "On a Bacterial Test for Estimating the Pollution of Air in Rooms and Inhabited Places."

Reports by Dr. Houston, "On the Chemical and Bacteriological Examination of the Tunbridge Wells Deep Wells Waters," and, "On the Nature and Relative Amounts of the Bacteria in the Contents of the Lower Bowel of the Healthy Human Subject." Speaking of these last-mentioned investigations, Dr. Houston finds that of the *Bacillus coli* group—the predominant organisms present—90 per cent. of the total microbes of that group are so markedly positive in their attributes as to preclude their being regarded as other than typical *B. coli*. The atypical *B. coli*, commonly found in sewages, effluents, etc., is rare in the contents of the lower bowel of the healthy human subject.

In a former report Dr. Houston had investigated the shallow wells of Chichester, and in this he speaks of the deep wells of Tunbridge Wells, which were found to be both chemically and bacteriologically of a high degree of purity. In a few instances coli-like microbes, were found isolated from a relatively large volume of water, which, however, could not, when duly tested, be confounded with the typical *B. coli* from the intestine. It is, however, a notable circumstance that the coli group of organisms is represented at all in water drawn from several hundred feet below the surface. Comparing the Chichester shallow wells with the Tunbridge Wells deep wells, 45 per cent. of the former yielded typical *B. coli*, whereas in no single instance was this organism found in the Tunbridge Wells waters.

Hitherto it has been assumed that bacteria in the air are disseminated mainly in the dry state. Dr. Mervyn Gordon's experiments afford examples of transference, which may be of frequent occurrence in the act of speaking, where the bacteria remain in the moist condition. They give a test capable of being used as an index of the pollution of air comparable with the tests for *Bacillus coli* as an index of the pollution of water.

As the result of the examination of the saliva of a number of healthy persons, he found that streptococci of various kinds are extremely abundant in human saliva, and in particular *Streptococcus brevis* is commonly present to the amount of at least ten millions per cubic centimetre of the secretion. Among other tests he found that this streptococcus gives a definite colour reaction with Rothberger's neutral-red broth when incubated under anaerobic conditions at 37° C., changing the red colour to a canary yellow with slight fluorescence. He proved that minute quantities of saliva produce the same effect. Dr. Gordon then applied this test to the air of a large room, first by placing Petri dishes charged with neutral-red broth set about the empty room, and afterwards by placing similarly another set of Petri dishes charged as before, set about the room during loud speaking by a person standing at one end of the room. He repeated these experiments with a series of different speakers. He thus was able to show, first, the absence of all streptococci before the speaking began, and afterwards to prove that droplets of saliva were diffused generally throughout the air of the room, even as far as 40 feet away from the speaker in front and 12 feet from him behind. In view of the fact that phthisical patients do cough up invisible droplets of mucus or saliva containing the material cause of their malady, Dr. Gordon's experiments afford a most valuable means of gauging the possible contamination of the air by the human subject. Dr. Gordon has also examined the air in different places in London and its vicinity by methods founded on these observations.

The water-supply at Bridgend, Glamorganshire, had for several years been tested both chemically and bacteriologically with uniformly satisfactory results, but in December, 1900, the bacteriological examination began to show doubtful characters, and more detailed and frequent examinations confirmed these doubts, though the chemical examination remained satisfactory. The water was tested both by the local analyst and by Dr. Thorpe at the Government Laboratory.

This water-supply is obtained from springs rising in a carboniferous limestone rock containing many fissures, and the collecting basin or area is liable, in times of heavy rain and in certain states of the tide in the tidal river adjacent, to occasional connection with polluted waters, and the practical outcome is to show that though the pollution may be small in amount, yet it may be dangerous in its quality or character, and that in such cases of unexplained illness the chemical analysis should be supplemented by a bacteriological examination.

Had time permitted, there are many other points of interest on which I might have spoken, but the preceding imperfect survey may, I hope, be useful to members. And now the time has come when I close my term of office, and give up this chair to a worthy successor. Mr. Bevan needs no introduction from me. He has already done yeoman service for many years as your Secretary, and though he has not been able to retire on the emoluments of that important office, yet I am sure all of us willingly accord to him heartfelt gratitude, and welcome his accession to the office of President. I wish him as pleasant a tenure of office as I have had. On my own behalf, I thank you all for the honour and the confidence you placed in me two years ago, and especially do I wish to thank the Secretaries, the Editor, and the Members of the Council, for their unvarying kindness to me.

I hope that the future may be ever bright for the Society of Public Analysts, and that the past record of good and useful work done by its members may be greatly surpassed in the years to come.

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THE INVERSION OF CANE-SUGAR IN PRESENCE OF MILK CONSTITUENTS.

BY THE HON. FRANCIS WATTS, C.M.G., D.Sc., F.I.C., AND H. A. TEMPANY, B.Sc., A.I.C.

(Read at the Meeting, February 1, 1905.)

IT was observed by one of us, while making analyses of condensed milks containing cane-sugar, that the method of Stokes and Bodmer gave results which were appreciably too low.

This method was described in the ANALYST, vol. x. The following description of the principles involved is taken from Allen's "Commercial Organic Analysis," vol. i., p. 343, 1898 edition:

"Coagulation of the milk by citric acid, dilution to ten times its original volume, filtration and titration of a portion of the filtrate with Pavy's ammoniacal copper solution. To 100 c.c. of the same filtrate 2 grammes of citric acid are added, the liquid boiled for ten minutes, cooled, neutralized, made up to 200 c.c., and titrated as before. The difference between the reducing powers of the solution before and after inversion is due to the invert sugar derived from the cane-sugar present, the milk-sugar not being inverted by boiling with citric acid."

It was observed that by following the process above detailed the complete inversion of the cane-sugar was not effected in presence of the constituents of milk. That this incomplete inversion was not due to insufficiency of citric acid was shown by a series of experiments in which 5 per cent. solutions of sucrose were boiled with 2, 1, and ½ per cent. of citric acid for ten minutes, and found to be fully inverted at the end of that time; it was therefore concluded that some constituent of milk exercised a retarding influence on the progress of the reaction.

To investigate this point a series of experiments was instituted whereby the progress of the inverting action of citric acid could be followed on two similar solutions of cane-sugar, one containing milk constituents, the other not.

The two solutions were prepared as follows:

Two portions of 100 c.c. each of a solution containing approximately 25 per cent. of sucrose were measured out, and to each of these 12 grammes of citric acid were added; to one solution 200 c.c. of whey, from which the curd had been precipitated by addition of a small quantity of citric acid, were added, and the two solutions then made up of 400 c.c. For further reference the solution containing milk constituents is termed A, and that not containing milk constituents B. The lactose in solution A was first determined by neutralizing the acidity of 25 c.c. by means of sodium hydrate, making up to 50 c.c., and determining by Fehling's solution.

The two solutions were then transferred to two flasks fitted with reflux condensers and with tubes whereby portions of liquid could be withdrawn without interfering with the progress of the reaction.

The two flasks were simultaneously heated up to the temperature of experiment, and from time to time portions of the contents withdrawn by means of the tubes provided. The portions withdrawn were rapidly cooled down to 30° C., 10 c.c. taken, neutralized with sodium hydrate, and made up to 100 c.c. with distilled water at 30° C.* The reducing sugars present were then determined by means of Fehling's solution.

Two series of experiments were performed. In the first series the two flasks were heated to 85° C. by immersing both in one large water-bath maintained at that temperature.

The following results were obtained:

Solution A, containing Cane-Sugar and Milk Constituents.			Solution B, containing Cane-Sugar only.	
Time from commencement of experiment at which solution was withdrawn.	Invert Sugar Per Cent.	Lactose Per Cent.	Time of withdrawing solution in minutes.	Invert Sugar Per Cent.
10 minutes	3·74	2·43	10 minutes	5·87
20 ,,	5·36	—	20 ,,	6·68
30 ,,	6·32	—	30 ,,	6·96
40 ,,	6·59	—	40 ,,	7·26
50 ,,	6·74	—	50 ,,	7·26
60 ,,	6·90	—	60 ,,	7·26
80 ,,	6·98	—	80 ,,	7·26

It will be observed that in the case of solution A the inversion was not quite complete in the time of experiment.

A second series of experiments was performed at the boiling-points of the solutions, the liquid in the flasks being kept at a gentle ebullition over a small flame.

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* 30° C. is the average laboratory temperature in the Leeward Islands, where these experiments were performed.

The following results were obtained :

Solution A, containing Cane-Sugar and Milk Constituents.			Solution B, containing Cane-Sugar only.	
Time from commencement of experiment at which solution was withdrawn.	Invert Sugar Per Cent.	Lactose Per Cent.	Time of withdrawing solution in minutes.	Invert Sugar Per Cent.
10 minutes	5·03	2·51	10 minutes	6·50
25 ,,	6·38	—	25 ,,	6·59
40 ,,	6·38	—	40 ,,	6·50
55 ,,	6·46	—	55 ,,	6·59
70 ,,	6·53	—	70 ,,	6·59
85 ,,	6·67	—	85 ,,	6·59
100 ,,	6·53	—	100 ,,	6·59
115 ,,	6·60	—	115 ,,	6·59

To ascertain definitely that citric acid is without action on lactose, as stated by Stokes and Bodmer, an experiment was performed in which 200 c.c. of whey were acidified with 12 grammes citric acid, made up to 400 c.c. with distilled water, and boiled as in the previous experiments, portions being withdrawn at equal intervals of time and the lactose estimated.

Time of withdrawal of solution in minutes.	Lactose Per Cent.
0 minutes	2·57
30 ,,	2·57
60 ,,	2·57
90 ,,	2·62
120 ,,	2·57

This experiment shows conclusively that, as stated by Stokes and Bodmer, citric acid is entirely without inverting action on lactose even after heating for a very considerable length of time.

From the results above detailed it is obvious that the presence of some constituent of milk exerts a marked retarding influence on the inversion of cane-sugar by citric acid, and that boiling for ten minutes, as advised by Stokes and Bodmer, is insufficient to insure complete inversion. To obtain satisfactory results the boiling should be continued for forty minutes.

On the electrolytic dissociation hypothesis the velocity of inversion of cane-sugar by acids depends on the active mass of the hydrogen ions present, reduction of the number of free hydrogen ions occasioning retardation of the reaction. This reduction, one would anticipate, would be occasioned by some dissociated substance in solution, which, interfering with the solubility product of citric acid, would cause reduction of the number of free hydrogen ions, and thus retardation of the reaction.

One would expect that the effect was not due to the presence of the lactose in solution, and that this is the case is shown by an experiment in which two similar solutions of cane-sugar, to one of which approximately 5 per cent. of lactose had been added, were boiled with 3 per cent. citric acid, portions being withdrawn and the reducing sugars determined from time to time as in the previous experiments.

Solution A. Cane-Sugar and Lactose.			Solution B. Cane-Sugar.	
Time of withdrawal in minutes.	Invert Sugar Per Cent.	Lactose Per Cent.	Time of withdrawal in minutes.	Invert Sugar Per Cent.
10 minutes	6·55	5·20	10 minutes	6·25
20 ,,	6·55	—	20 ,,	6·65
35 ,,	—	—	35 ,,	6·55
60 ,,	6·55	—	60 ,,	6·60

These results show, as one would anticipate, that lactose is entirely without influence on the rate of inversion of cane-sugar.

The practical bearing of these experiments appears to be that, in following the method of Stokes and Bodmer, ten minutes' boiling is insufficient to effect the complete inversion of all the cane-sugar present, and a longer time should be allowed for its completion. Forty minutes' boiling would appear sufficient to effect the inversion of all the cane-sugar present, if the milk under examination has been diluted down till it contains between 5 and 10 per cent. of cane-sugar.

In the foregoing experiments the determinations were made by titrating the solutions against 10 c.c. of Violette's modification of Fehling's solution diluted with its own bulk of distilled water. This method gives results sufficiently accurate for the purposes of comparison required in this inquiry ; for analytical purposes, where high accuracy is desired, it is preferable to use the gravimetric method as laid down by Brown and Morris.

GOVERNMENT LABORATORY,
LEEWARD ISLANDS, B.W.I.

DISCUSSION.

The CHAIRMAN (Mr. Blount) said that, as far as he had been able to follow the paper without having read it beforehand, the authors seemed doubtful as to what effect would be produced on the activity of the citric acid by a substance like lactose. Clearly, however, on the dissociation hypothesis, lactose, which was not an electrolyte in aqueous solution, could have no influence at all. The rapidity of inversion must depend on the activity of the citric acid. That, in turn, must depend on the degree of dissociation, which, again, depends on the degree of dilution and the nature of the solvent. This small criticism, however, did not invalidate the authors' general argument.

Dr. THORNE said that he might perhaps add to the paper a few words of explanation. The authors had sent it to him with the request that it might be published as he deemed most suitable, and it seemed to him to be undoubtedly a paper which should be laid before that Society. He had taken an opportunity of first reading through the paper himself, and he thought that, when it came to be printed, it would be found not to justify the Chairman's criticism. The authors' experiments with lactose were merely for the purpose of obtaining definite proof of what, as the Chairman had indicated, was *a priori* to be expected. The main question was that of the determination, in preserved milk, of quantities of cane-sugar which were small relatively to those of milk products. The method of Stokes and Bodmer seemed under such circumstances to give too low results, and the authors had endeavoured to ascertain whether better results could be obtained by increasing the time of inversion. Their experiments indicated that in the milk constituents there was something, the nature of which they had not yet been able to determine, which had a retarding influence on the inversion of the cane-sugar by citric acid. When the cane-sugar was relatively small in quantity, the retardation was sufficient to impair the accuracy of the method, and it was necessary to increase the time of inversion to forty minutes in order to obtain satisfactory results.

Mr. BAKER expressed surprise that the authors had not tried the use of invertase as an inverting agent for the cane-sugar. It had no influence on the lactose, even at the inverting temperature of 50° to 55° C., and its use would, as it seemed to him, get over all the difficulties the authors had mentioned.

Dr. SCHIDROWITZ thought that anyone looking at the matter from a purely chemical point of view would at once dismiss the idea that lactose could, under the conditions of this process, have any influence on the inversion of cane-sugar. It afforded an excellent illustration of the disadvantages of the system, or, rather, fashion, which had sprung up of regarding all chemical phenomena from the purely ionic standpoint.

Mr. J. B. P. HARRISON said that in a recent paper before the Society (ANALYST, 1904, xxix., 248) he had described a polarimetric method for the determination of cane-sugar in sweetened condensed milk. The specific rotatory power was determined before and after inversion in an aqueous solution of the condensed milk from which the fat and proteids had been removed by precipitation with acid mercuric nitrate. An excess of this reagent was used, and the inversion was brought about by the action of the excess on boiling a portion of the filtrate for seven minutes.

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THE COLORIMETRIC ESTIMATION OF SALICYLIC ACID IN FOODSTUFFS.

By FRED T. HARRY and W. R. MUMMERY.

(*Read at the Meeting, February* 1, 1905.)

The work was carried on with a view to obviating the difficulties experienced in obtaining a good colour with Fe_2Cl_6, when using the present methods employed for estimating small quantities of salicylic acid in foodstuffs containing tannin bodies.

The test is based upon the fact that lead tannate is insoluble in caustic alkalies, while lead salicylate is readily soluble, and resolves itself into the separation of the lead salts of both acids by means of potash or soda.

The process which gave the best results with preserved fruits, fruit pulps, or jams is the following: Fifty grammes of the crushed sample to be tested are placed in an ordinary flask which has previously been marked at 300 c.c.; a small quantity of water is added, then 15 to 20 c.c. of a saturated solution of basic lead acetate, and the whole made alkaline by the addition of about 25 c.c. of roughly normal soda.

The alkali first throws down the excess of lead, afterwards dissolving the hydroxide and a quantity of albuminoids, etc., which are reprecipitated on adding acid, giving a clear colourless filtrate; to effect this, 15 to 20 c.c. roughly normal HCl are added.

The above procedure was found necessary in order to obtain complete solution of the lead salicylate, which is not reprecipitated by the acid.

The contents of the flask are then made up to the 300-c.c. mark, well shaken, and filtered cold through Rhenish paper, 200 c.c. of the filtrate being collected. The filtrate is acidified with HCl, refiltered if necessary, and extracted three times with ether; HCl has been used in preference to H_2SO_4, as lead chloride is easily filtered off and washed, and if present in small quantities only, does not interfere with the extraction.

The ether is distilled off, the salicylic acid dissolved in a small quantity of dilute alcohol, made up to 100 c.c., and estimated colorimetrically with Fe_2Cl_6 in the usual way.

The result multiplied by three will bring the amount to percentage.

The presence of alcohol in the liquid to be extracted prevents complete solution of the salicylic acid in ether; in cases where large quantities of alcohol are present this must be driven off before proceeding with the test. To effect this, the sample must be made alkaline and boiled gently; afterwards the process is continued as before. It is advisable to neutralize the alkali before adding lead acetate, or too great an excess of the former may result.

Different samples require different treatment (with regard to the quantity of lead acetate and alkali used), in order to insure the maximum rapidity of filtration combined with the smallest quantity of lead hydroxide dissolved; there is one point at which this takes place, a point which can readily be judged by practice.

If the contents of the flask are made too alkaline, filtration is rapid, but a large excess of lead and albuminous bodies come through into the filtrate, making a second filtration necessary; on the other hand, if too acid, filtration is also rapid, but the complete solution of the lead salicylate is then doubtful. After the addition of the lead acetate and alkali the contents of the flask may be raised to boiling with safety; this will materially assist filtration, but has the disadvantage of allowing a large amount of lead to go through into the filtrate. We do not recommend this unless absolutely necessary; in any case, the filtration should be conducted in the cold.

For beers the process used was the following: 100 c.c. of beer are placed in a flask marked at 200 c.c., made alkaline with 5 c.c. roughly normal NaOH, and all the alcohol driven off; this should be done at a temperature just below the boiling-point, as vigorous boiling appears to result in loss even when the solution is alkaline.

After cooling, 5 c.c. of roughly normal HCl are added, and then at least 20 c.c. basic lead acetate; the whole is then made alkaline with about 20 c.c. normal NaOH, and made up to the mark. At this stage the whole may be raised to boiling and allowed to cool before filtering, but this may be omitted if thought advisable.

One hundred c.c. of the filtrate are collected and acidified as before; in this case a precipitate of lead chloride is thrown down, which is filtered off, and the filtrate extracted with ether as before.

The process which gave the best results with wine was the same as with beer—taking 50 c.c. of the wine, making up to 100 c.c., and filtering off 50 c.c. As with beer, it was necessary to add a large excess of lead acetate, and to have the whole fairly alkaline, in order to exclude all tannin bodies from the filtrate.

If any tannin colours present themselves in the process of matching, they are probably due to using an insufficient quantity of lead acetate.

The lead acetate used was a saturated solution made in the usual manner by boiling with litharge.

The tendency which beers and wines have to emulsify when shaken with ether is entirely obviated by this process, it being impossible to produce an emulsion with the filtrate even with the most vigorous shaking.

With regard to the actual colorimetric readings, these were observed in ordinary 50-c.c. Nessler tubes, using a very weak solution of ferric chloride, which should be made up fresh when required. The standard salicylic acid solution used was 0·01 per cent., which could be kept without change for a very long time.

In a paper read before this Society on November 5, 1902, Mr. Harvey recommended the use of ferric alum instead of ferric chloride, claiming that it gives with salicylic acid a deeper and more permanent colour. We have tried several samples of ferric alum, and are unable to confirm Mr. Harvey's results.

On allowing two tubes to stand over night, one containing ferric alum and the other ferric chloride, we found that the former had become quite turbid and the iron had separated out, while the latter, although not the same tint as at first, was just as clear.

The use of sulphuric acid in the ferric alum solution as recommended is also a drawback, since using one drop in excess will prevent the colour with salicylic acid appearing at all.

We find our process gives excellent colours with black beers and wines containing large quantities of tannin, and works perfectly satisfactorily, even after the addition of about 0·5 gramme tannic acid in solution.

The subjoined table will show the degree of accuracy which may be expected:

Sample.	Salicylic Acid added in Grains per Pound or per Pint.	Salicylic Acid found in Grains per Pound or per Pint.	Error.
Apple pulp	0·23	0·22	0·01
"	0·45	0·36	0·09
"	0·45	0·43	0·02
"	0·45	0·32	0·13
"	1·06	0·93	0·13
"	1·50	1·36	0·14
Blackberry jam	0·40	0·30	0·10
Apple pulp	0·70	0·54	0·16
Blackberry jam	0·56	0·42	0·14
Black currant jam	3·80	3·77	0·03
Preserved apricot	3·80	3·77	0·03
Bitter beer	1·14	0·91	0·23
"	1·14	0·92	0·22
"	1·14	0·91	0·23
Preserved apricots	1·50	1·40	0·10

The working error of the method applied to beers, etc., appears to be slightly larger than in the case of fruit pulps, jams, etc., but this is no doubt due to the boiling required at the commencement of the operation and the subsequent filtration. Again, any method that depends on an extraction with ether and a colorimetric matching must of necessity possess an inherent minus error. In the case of jams, etc., the average error is about 0·09 to 0·1 grain per pound, while in the case of beers, etc., it is about 0·22 grain per pint.

We may, perhaps, point out that we have in all cases worked with quantities representing about 1 grain per pint, or 1 grain per pound in the case of solids, partly because in the estimation of larger quantities the percentage error due to the interference of tannin bodies is very much smaller, and such cases consequently present no difficulty. We have also been influenced by the consideration that the above-mentioned quantity is the limit recommended by the Preservatives Committee.

In conclusion, we should like to take this opportunity of thanking Mr. Chapman for his kindness in not only allowing us the use of his laboratory, but also for the personal assistance he has given us in carrying out this work.

DISCUSSION.

The CHAIRMAN (Mr. Blount) having invited discussion,

Mr. BEVAN said that the difficulty of getting the salicylic acid in a state of purity was well known to those who were in the habit of estimating it, either directly or by titration or colorimetrically, and they were therefore much indebted to the authors for showing how that difficulty might be overcome. The authors had mentioned, in the case of beer, an error of 0·23 in 1·14, or something like 25 per cent. He should like to hear whether, supposing the quantity of salicylic acid present to be 11·4, the error would be 2·3. If so, it would be a rather serious error.

Dr. SCHIDROWITZ said that in his experience there was an exaggerated view as to the frequency of the occurrence of salicylic acid in beer. He should like to know whether the authors had ever met with a sample of beer which did contain salicylic acid; he himself never had.

Mr. BEVAN: In the case of lager beer, certainly.

Dr. SCHIDROWITZ said that some little time ago attention had been strongly directed to the use of salicylic acid in imported beer, and he had taken the trouble to obtain samples of nearly every kind of lager beer in London, but had not been able to detect salicylic acid in any of them. If, however, beer or wine containing salicylic acid was kept for some time, the salicylic acid undoubtedly disappeared gradually.

Mr. CHAPMAN said that the paper to which they had listened that evening had had its origin in the fact that in his laboratory it had been found absolutely impossible to detect salicylic acid in one or two samples of fruit pulp in which it was known to be present in small amounts. Some fruits contain relatively large quantities of tannin and other bodies capable of producing a dark coloration with ferric chloride, and this in some cases entirely masks the fainter colour due to the salicylic acid. It was with the object of surmounting this difficulty that the authors had tried the method now described, and had found it to be very satisfactory. The method worked well with beer, but, as in the case of most analytical processes, some experience of it was essential for its successful application. A very interesting point in connection with the method was that the solutions to be extracted showed no tendency whatever to emulsify. This was, of course, highly important, inasmuch as the formation of emulsions is one of the most fruitful sources of error in processes involving extraction by the employment of immiscible solvents.

Mr. A. E. PARKES said that he had not quite followed the way in which the authors treated the ether extract. If the solvent were distilled off, the amount of salicylic acid obtained would be lower than when the ether was extracted with alkaline water or allowed to evaporate spontaneously.

Mr. LING inquired whether a clean crystalline residue was obtained after the first extraction, or whether it was necessary to extract again. In the ordinary process one had to make two extractions, one with ordinary ether and one with petroleum spirit, in order to obtain anything like a clean residue.

Mr. HARRY, in reply, said that they had only tried the method with small quantities of salicylic acid, but were certainly of opinion that with larger quantities the error would not be larger in proportion. There was no great difficulty in estimating large quantities of salicylic acid in beer, as the tannin was but small in quantity and did not interfere; in fact, they doubted whether it would be necessary to use this method at all. With regard to the treatment of the ether extract, they distilled off the ether in an ordinary Soxhlet apparatus, and then made up with dilute alcohol, the estimation being proceeded with as described. In some cases the residue might not be quite dry, but where a fairly dry extract was obtained it was quite crystalline and generally colourless. In some beers it might be slightly yellow, but this was not noticeable when it was in solution.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Influence of Various "Fining" Materials on the Composition of Wine. K. Windisch and T. Roettgen. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 129-133.)—The authors have previously described the action of casein, milk, and charcoal on wine (ANALYST, 1904, 370), and now give the results of their experiments with isinglass, gelatin, white of egg, and "Spanish earth," the latter being a disintegration product of felspar. Except in the case of white of egg, which was found to increase the total solids and mineral matter in red wine, none of these fining materials had any influence on the acidity, total solids, or mineral matter in red or white wine. As was to be expected, gelatin and white of egg decreased the amount of tannin in the wines, as did also the "Spanish earth." The nitrogenous matters in the wines remained unaltered in quantity in all the experiments. The general conclusion arrived at is that the composition of wine is but little influenced or changed by any of these fining materials. It might be mentioned that some samples of "Spanish earth" contain considerable quantities of calcium carbonate, and are quite unsuitable for clarifying wine, as they neutralize the acidity of the latter.

W. P. S.

Fat Extraction from Milk. Th. Thomsen. (*Tidskrift for Landocconomi*, February, 1905, 101.)—The usual methods of extracting fat from milk after drying on paper, gypsum, etc., have, as is well known, failed in giving correct results in the cases of skimmed milk, butter-milk, or milk which has undergone violent agitation. In such cases the Gottlieb-Röse method (ANALYST, 1898, xxiii., 259, and 1904, xxix., 112) gives higher results. It has been supposed by several analysts that the low results obtained in the former method were due to the retention of a portion of fat, viz., the very minute globules, which had become incorporated with the fat solids, and to ascertain if this was the cause the author has made the following investigations. He found that by treating the sample of milk with pepsin and hydrochloric acid before drying, the thorough peptonization caused the milk solids to dry in a loose form, which insured a complete extraction. In this way he obtained absolute agreement between the results obtained by extraction and by shaking out in the Gottlieb method.

H. F.

The Influence of Formalin on the Estimation of Total Solids in Milk. H. Höft. (*Chem. Zeit.*, 1905, xxix., 54.)—Some experiments were performed in order to test whether the addition of formalin to milk made any appreciable difference in the weight of total solids, and, if so, what the exact difference was.

The author gives figures which show, both for full and for separated milk, that an addition of any amount of formalin below 0·6 c.c. per 100 c.c. of milk has no effect on the total solids, even after twenty-five days. As the amount of formalin usually added for the purpose of preserving milk is said to be 4 drops per 100 c.c., this amount can have no effect on the analysis of the milk as regards total solids. The addition of 0·6 c.c. makes a difference of about 0·1 per cent. in the total solids, and increasing amounts of formalin have a corresponding influence on the figures.

E. K. H.

The Adulteration of Beeswax. R. Cohn. (*Zeit. f. Off. Chem.*, 1904, x., 404.)—The author disagrees with the usual methods for obtaining the saponification number of substances saponifiable with difficulty, such as beeswax, and especially with the limited time allowed for saponification. From a series of 200 experiments he considers that the saponification number for this substance may occasionally rise as high as 108, though usually it is about 100. These values could, however, rarely be obtained by treating for only one hour, as usually recommended, certain samples of pure wax requiring six hours. To this cause must be attributed the fact that many samples of pure wax are reported to be adulterated.

The method suggested is the following: 2 to 3 grammes of the wax are dissolved in 25 c.c. of 96 per cent. alcohol, 25 c.c. $\frac{N}{2}$ KOH (in 95 per cent. alcohol) added, and boiled for three hours under a reflux condenser. The excess of KOH is titrated with $\frac{N}{2}$ HCl and using phenolphthalein as indicator, the acid being added until the red colour no longer reappears on boiling.

Saponification with NaOH is very much slower, and this the author attributes to the different solubilities of the potassium and sodium compounds formed.

The maximum values obtained averaged 102, the minimum 96, whilst the greater number of the experiments averaged 100. This (100) is therefore suggested as the standard saponification number, and is the factor employed in the calculation of three typical analyses, details of which are given.

H. A. T.

Oil of Eucalyptus Polybractea. John C. Umney and C. T. Bennett. (*Pharm. Journ.*, 1905, lxxiv., 143.)—This species of eucalyptus is that known as the "Blue Malee," and is very closely allied to *E. dumosa.* An authentic specimen of the oil from *E. polybractea* has recently been examined by the authors, with the following results: Specific gravity, 0·929; optically inactive; cineol, as determined by Schimmel's process, 79 to 80 per cent. About 78 per cent. of the oil distilled between 173° and 176° C. The oil is quite free from any irritating odour of aldehydes, and contains only very small amounts of aromadendral and cumic aldehyde. In this respect the oil differs from those from the same source examined by R. T. Baker ("Research on the Eucalypts," p. 116), who states that the oil resembles that from *E. cineorifolia*, which contains aromadendral in quantity.

If the authors' sample of the oil from *E. polybractea* be a normal distillate, it is of great medicinal value, and in this respect is not even second to the official oil from *E. globulus.* The species grows luxuriantly, and the oil should be obtainable in large quantities.

A. R. T.

Adulteration of Eucalyptus Oil with Castor Oil. C. T. Bennett. (*Chemist and Druggist*, 1905, 33.)—Extensive adulteration of commercial eucalyptus oils with castor oil, to the amount of 12 to 20 per cent., has recently been observed. The adulterated oils, which complied with the tests of the British Pharmacopœia, had the following characters: Specific gravity, 0·917 to 0·919; optical rotation, 0° to + 2°; cineol, 38 to 45 per cent. Suspicion was aroused owing to the low cineol content and comparatively high specific gravity. On fractional distillation, 80 per cent. of the oil distilled over, leaving a viscid, non-volatile portion in the flask. This fraction had a specific gravity of 0·957 and a refractive index of 1·4810. It absorbed a large proportion of caustic potash on saponification, and glycerin was present in the liquid. The adulterant was soluble in 90 per cent. alcohol and in petroleum spirit, but when excess of the latter solvent was added, separation took place. These reactions are characteristic of castor oil. On destructive distillation, a sample of castor oil yielded a fraction having precisely similar characters to the product obtained from the adulterant in the same way (specific gravity, 0·866; 0·869; refractive index, 1·4345; 1·4360). This limpid and pungent liquid contained in each case aldehydes and acrolein. An admixture of 5 to 10 per cent. of castor oil in eucalyptus oils of good quality may easily be overlooked unless special tests are applied. A. R. T.

A Contribution to the Knowledge of Arachis. W. Moser. (*Chem. Zeit. Rep.*, 1905, xxix., 6.)—"Earth-nut" or "ground-nut" cakes, made from *Arachis hypogæa* freed from oil, are largely used as food for cattle. The author describes the preparation and properties of an alkaloid isolated by him from the earth-nut, in addition to the bases cholin and betain previously found therein by E. Schutze. The author suggests the name "Arachin" for this new substance. Its poisonous action was investigated by subcutaneous administration to animals, but the tests were inconclusive, and further experiments are necessary. As the author always obtained this "arachin" in the numerous samples of earth-nut meal investigated, he comes to the conclusion that it exists already formed in the fresh seeds, and is not formed as a decomposition product of the proteids during storage.
E. K. H.

The Detection of Gum Arabic in Gum Tragacanth. E. Payet. (*Ann. de Chim. anal.*, 1095, x., 63.)—A distinctive test is based on the fact that gum arabic contains an oxydase while gum tragacanth does not. A solution of the gum (1 : 30) made in the cold is shaken with an equal volume of a 1 per cent. aqueous solution of guaiacol and 1 drop of hydrogen peroxide solution. If gum arabic be present the liquid rapidly turns brown, but remains absolutely colourless if the gum tragacanth was pure. C. A. M.

ORGANIC ANALYSIS.

Determination of Glycerol in Soap. E. Martin. (*Moniteur Scient.*, [4], xvii., 797; through *Pharm. Journ.*, 1904, lxxiii., 958.)—Ten grammes of the soap are dissolved in 50 c.c. of hot water, the fatty acids liberated from the solution by the addition of a slight excess of diluted sulphuric acid, and the solution kept hot until the acids are completely melted. The liquid is filtered through a wetted filter-paper, and the fatty acids washed on the filter with boiling water. Excess of basic lead acetate solution is added to the filtrate, the liquid filtered after half an hour, and the precipitate washed. The excess of lead in the solution is then precipitated by the addition of a little sulphuric acid, and the liquid made up without filtration to 250 c.c. When the precipitate has subsided, 25 c.c. of the clear solution (=1 gramme of soap) are examined by Hehner's dichromate method for the determination of glycerol in the usual way, whereby the glycerol is quantitatively oxidized to carbon dioxide. This gives the free glycerol. If the combined glycerol be also required, as in the case of superfatted soaps, a further determination of the glycerol after saponification must be made. The difference between the total glycerol thus found and the free glycerol gives the proportion of combined glycerol in the sample. A. R. T.

A New Sugar from Mountain Ash Berries. G. Bertrand. (*Bull., Soc. Chim.*, 1905, xxxiii., 166-168.) When the juice of these berries, or, better still, the mother-liquid from the crystallization of sorbitol, is fermented with sorbose bacteria, there remains in the liquid a substance that can be isolated as a benzoic acetal, and which Vincent and Meunier regarded as an octitol. The author, however, has been able to separate this substance in crystalline form, and shows that it is a hexitol of the formula $C_6H_{14}O_6$. He gives it the provisional name of *sorbierite.* The new sugar crystallizes in anhydrous rhombic plates, which are extremely soluble and deliquescent, and melt at 73·5° C. It is lævorotatory, an aqueous 10 per cent. solution at 20° C. giving $[\alpha]_D = -3° 53$. Its molecular weight determined by the freezing-point method was 174·2. It does not reduce Fehling's solution. It combines with benzaldehyde in the presence of sulphuric acid to form a mixture of two acetals, one of which melts below 190° C., and is readily soluble in boiling alcohol, while the other melts about 242° C., and is much less soluble in alcohol. C. A. M.

Artificial Colouring Matter in Ceresin. Utz. (*Oest. Chem. Zeit.*, 1904, vii., 509.)—From the examination of a number of samples of commercial ceresin, the author finds that the yellow varieties are invariably coloured with coal-tar dyes. He considers such admixtures to be distinctly injurious. H. A. T.

Note on Halphen's Reaction for Cotton-seed Oil. K. Fischer and H. Peyau. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 81-90.)—The authors agree with previous observers that cotton-seed oil, when heated to a temperature of 250° C., no longer gives a coloration with Halphen's reagent (*see* ANALYST, 1899, 214; 1900, 270; 1903, 79). When heated to this temperature, however, the oil becomes quite unedible. Further experiments showed that, by treating the oil with zinc and sulphuric acid, no weakening of the reaction is produced. Bleaching by chlorine, while rendering the oil unfit for food, completely destroys its sensibility to Halphen's reagent. Treatment with sulphurous acid, followed by washing with alcohol to remove acid products formed, yields an oil which gives no coloration with this test, and which cannot, in other respects, be distinguished from natural cotton-seed oil. The presence of small quantities of cotton-seed oil so treated in other oils can, therefore, only be detected by means of the phytosterol-acetate test (ANALYST, 1902, 94). W. P. S.

The Detection of Foreign Oils in Walnut Oil. J. Bellier. (*Ann. de Chim. anal.*, 1905, x., 52, 53.)—In many districts of France walnut oil is preferred to olive oil for food purposes, and as its price is often more expensive than that oil, it is very liable to adulteration. Most of the ordinary oils can be detected by the usual methods, but a mixture of equal parts of walnut and poppy oils is hardly to be distinguished from the pure oil. One difference between the two oils is the freezing-point, walnut oil solidifying at - 27·5° C., and poppy oil at - 18° C., but a test based on this difference is not easy to apply. The author has therefore devised a test based on the difference in solubility of the respective solid fatty acids in alcohol (about 70 per cent.) containing potassium acetate. The solutions required are (1) 25 c.c. of glacial acetic acid with 75 c.c. of water; and (2) 16 grammes of pure potassium hydroxide in 100 c.c of 91 to 93 per cent. alcohol. The amount of solution (1) required to neutralize 5 c.c. of solution (2) should be determined. Usually it is about 2·5 c.c. In testing the oil, 1 c.c. is mixed with 5 c.c. of the alkali solution (2), and a parallel test made with walnut oil of known purity. The tubes are heated below the boiling-point until the oils have dissolved, and are then closed and placed for thirty minutes in a water-bath at about 70° C., after which the quantity of the acetic acid solution (1) corresponding to the 5 c.c. of alkali solution is introduced, and the tubes again closed and kept in water at 17° to 19° C., with frequent agitation. Under these conditions walnut oil requires a long time to produce even a scanty precipitate at the bottom of the tube, whereas poppy oil speedily yields an abundant one, which remains distributed throughout the liquid in the tube. Other oils give an immediate abundant precipitate, the contents of the tube becoming nearly solid. In this way it is possible to detect a very small percentage of olive, sesamé, cotton-seed, arachis, linseed, or rape oils in walnut oil. In the case of poppy oil the results are uncertain with less than 10 per cent., but with 15 to 20 per cent. there can be no doubt.

The following values were obtained in the analysis of walnut, poppy, and edible linseed oils:

	Walnut Oils.			Poppy Oil, Levant.	Edible Linseed Oil.
	Cold Pressed.	Cold Pressed.	Hot Pressed.		
Specific gravity... ...	0·9256	0·9253	0·9255	0·924 to 0·9235	0·930
Iodine value	145·3	145·8	145·9	138 to 139·9	159·8
Maumené test... ...	101° C.	100° C.	101° C.	100° to 99° C.	130° C.

C. A. M.

Note on Bergamot Oil and other Oils of the Citrus Series. Herbert E. Burgess and Theodore H. Page. (*Proc. Chem. Soc.*, 1904, xx., 181.)—Amongst the minor constituents of oil of bergamot, acetic acid, pinene, camphene, limonene, and octylene have been detected. The pungency of the first fractions of bergamot oil is due to acetic acid, which occurs in smaller quantities in lemon oil, and which is no doubt present in other oils of this class.

The octylene present in oil of bergamot is identical with that present in lemon oil, since, on oxidation with permanganate, butyric acid is formed in each case. Thus octylene is probably a normal constituent of all citrus oils. A second phenylurethane has been isolated from the terpineol-containing fraction of distilled oil of limes. This compound is more soluble than that obtained from ordinary terpineol, crystallizes in tufts of needles, and yields on hydrolysis an oily body having a strong odour of oil of limes. A. R. T.

Characteristics of Certain African Copals. C. Coffignier. (*Bull. Soc. Chim.*, 1905, xxxiii., 169-176.)—*Kissel Copal.*—This consisted of small, yellowish-red, vitreous fragments, without odour, and very free from impurities. It became soft at about 65° C., and melted at 110° C. It dissolved in sulphuric acid of 66° B., (S.G. 1·843) yielding a dark-brown solution and a chocolate froth on shaking. On treatment with boiling alcohol it left 57·4 per cent. of a hard, brilliant residue, while 42·6 per cent. of friable yellow residue was obtained on extraction with ether. It gave the following values: Specific gravity, 1·066; acid value, 70·4; and saponification value, 117·8.

Cameroon Copal.—This was in rounded, irregular lumps of a pale-yellow or reddish-yellow colour, and with an odour recalling that of pepper. It had a brilliant fracture, and was very hard. When heated it softened about 100° C., and melted at 150° C. Sulphuric acid dissolved it at once, giving an orange-yellow solution, becoming red after twenty-four hours. On shaking the liquid a deep yellow froth was obtained. A friable residue (66·7 per cent.) was obtained with boiling alcohol, while ether left a yellowish-white residue of 55·8 per cent. Analytical values: Specific gravity, 1·052; acid value, 159·7; and saponification value, 70·0 (?170·0).

Accra Copal.—Irregular pieces of yellowish-white colour, vitreous or opaque, with a strong pepper-like odour. On heating it softened at 75° C., and melted at about 120° C. Sulphuric acid dissolved it, giving a reddish-brown solution, which frothed red on shaking. Alcohol left 47·8 per cent. of a pale-yellow, friable residue, while ether left 44 per cent. of a similar residue. Analytical values: Specific gravity, 1·033; acid value, 97·8; and saponification value, 140·0. C. A. M.

The Detection and Determination of Bituminous Substances in Gutta-percha. Pontio. (*Ann. de Chim. anal.*, 1905, x., 57, 58.)—Gutta-percha is stated to be frequently adulterated with bituminous substances, and the following method is recommended for their determination. Bitumen is composed mainly of a mixture of liquid petrolene, $C_{20}H_{32}$, and of asphaltene, $C_{20}H_{32}O_3$, with some sulphur compounds. It is soluble in petroleum spirit, toluene, benzene, chloroform, and carbon bisulphide, and is partially soluble in ether (38 per cent.) and alcohol to the extent of 7 per cent., the resinous substance dissolving in alcohol being also soluble in ether. On the other hand, gutta-percha is completely soluble in boiling ether, and thus, on extracting a mixture of gutta-percha and bitumen with that solvent, 62 per cent. of the latter will be left in the insoluble residue. In examining a commercial sample it is first digested with absolute alcohol for the determination of the resinous constituents, and is then extracted for twelve hours with boiling ether, after which the residue is dried and weighed. This residue is then treated with boiling carbon bisulphide or toluene, which will dissolve the 62 per cent. of bituminous constituents if present, and the residue from this extraction will consist of natural impurities in the gutta-percha. C. A. M.

The Quantitative Determination of Betaine. V. Staněk. (*Zeit. Zuckerind in Böhmen*, xxviii., 578-583; *Biochem. Centralbl.*, 1905, iii., 471.)—Betaine is precipitated by a solution of iodine in potassium iodide, yielding brownish-red crystals, rapidly changing to green, which are readily soluble in alcohol, hydriodic acid, and an aqueous solution of potassium iodide, but dissolve with difficulty in water. The compound, which has the formula, $C_5H_{11}NO_2.HI.I_5$, melts at 58° to 61° C., with evolution of iodine, and is decomposed by boiling water into iodine and hydrated betaine iodide. The best reagent for the quantitative precipitation of betaine consists of a solution of 100 grammes of potassium iodide and 153 grammes of iodine in 200 c.c. of water. The solution of betaine, the strength of which should be from 1 to 4 per cent., is treated with a little sodium chloride or sulphuric acid, to reduce the solubility of the betaine hexaiodide, and an excess of the reagent introduced. After standing for three hours, the precipitate is collected in a Gooch crucible, and washed with water. It is then transferred to a Kjeldahl flask, where it is decomposed with sulphuric acid, and the liberated iodine collected in a flask containing alcohol. The residual liquid in the flask is boiled until colourless, after the addition of mercury, the nitrogen determined in the usual manner, and the amount of betaine calculated from the result. In this way betaine can be quantitatively determined in the presence of glycocoll, asparagine, tyrosine, and ammonium salts. C. A. M.

New Reaction for Enzymes. Winckel. (*Pharm. Post*, xxxvii., 598; through *Pharm. Journ.*, 1904, lxxiii., 957.)—The enzymes emulsin, myrosin, rennet, diastase, trypsin, invertin and ptyalin all give a violet colour when treated with vanillin and hydrochloric acid, and this reaction is suggested as a method for the detection of enzymes. The coloration is readily obtained by treating sections of fatty and oily seeds containing enzymes with the above-named reagents. A. R. T.

A Method for the Rapid Ultimate Analysis of Certain Organic Compounds. John Norman Collie. (*Proc. Chem. Soc.*, 1904, xx., 174.)—The combustion of the substance is carried out in the presence of a known volume of oxygen, and any change in the volume of the gas subsequently noted. The carbon dioxide produced is then determined by absorption in the usual manner and, from these data, the percentage of carbon and hydrogen in the compound may be calculated.

The method is applicable only to those substances which are not appreciably volatile in a vacuum, and which contain only carbon and hydrogen, or these two elements combined with oxygen. The method is capable of being used in cases where only small amounts of substance are available, and it is very accurate, since the determinations depend upon the measurements of volumes of gases. An analysis can be made in about one hour. A. R. T.

Simplified Ultimate Organic Analysis: Quick Method. Communication from the State Chemical Laboratory in Hamburg. M. Dennstedt. (*Chem. Zeit.*, 1905, xxix., 52.)—The author has attempted to simplify the process of estimating the elements in organic compounds so that constant attention is unnecessary, and thus save time for other work, rather than to shorten the time taken by the single analysis.

The process used is a modified form of that already described by the author ("Anleitung zur vereinfachten Elementaranalyse," Otto Meissner's Verlag, Hamburg), and is not, therefore, detailed in the present communication, a knowledge of its contents being pre-supposed. The principle involved is that the substance should be largely volatilized in oxygen and carried over platinized quartz, where the combustion proper takes place.

Difficulty was encountered in getting this combustion to take place evenly, as the inflammable gases produced by volatilization formed an explosive mixture with the oxygen. To remedy this, the substance to be burnt was placed in an inner tube, and only a limited supply of oxygen allowed to pass over it, the main bulk of oxygen passing between the tubes, and only meeting the inflammable gas at the platinized quartz. Various devices were tried and rejected, for details of which reference must be made to the original paper, but the following account of the final process is of interest:

A combustion tube prepared and dried as usual is used: about the middle is a piece of platinized quartz, in front of which, in the case of sulphur or halogen-containing substances, are four small or two large boats filled with pieces of quartz coated with lead peroxide—the latter prepared by covering pieces of quartz of the size of a pea with a coating of a thin paste of lead peroxide and water, and drying in an air-bath at 100° C.

The front end of the tube is connected to the requisite absorption apparatus, and the platinized quartz heated to redness.

The substance is weighed in a boat, if solid, in a bulb, if liquid, and placed in the inner tube, the front end of which is open, and the back end either entirely closed for very volatile substances, or more usually drawn out to a fine capillary. A piece of previously ignited quartz is put next the boat (or bulb), and then three small pieces of platinum quartz. This tube is pushed down the combustion tube till the open end is close up to the platinized quartz. A well-fitting cork with a tube for passing in oxygen is then inserted in the combustion tube.

The heating is next started from the rear forwards, whilst a stream of oxygen is passed in. The rate of heating and of the oxygen must be regulated, so that a small flame is just maintained on the platinized quartz. If the volatilization is too great, carbon, or unburnt substance is carried beyond the platinized quartz, and so not burnt.

After the disappearance of the flame, which marks the end of the combustion proper (this takes from fifteen to twenty minutes usually), any carbon deposited on the boat and inner tube is burnt off by raising the temperature through the full power of the burners.

In order to see the flame above-mentioned, one of the top tiles is provided with a mica window.

One analysis can be quickly followed by another, as the tube will have cooled sufficiently for the introduction of the second substance in the time taken to change the absorption apparatus.

In this quick process it is necessary to renew the first soda-lime tube comparatively soon, but warning is given by the second tube increasing unduly in weight.

The author has successfully used this form of apparatus for the analysis of a great variety of substances, including such diverse things as carbon disulphide, which it has been found impossible to analyse by the usual combustion methods, ether, alcohol, acetanilide, phenanthrene, oils, caoutchouc and factis, these last two substances, which are exceedingly difficult to manipulate by the older methods, give no trouble with the author's method. E. K. H.

INORGANIC ANALYSIS.

Testing Zinc Oxide used for the Estimation of Manganese by Potassium Permanganate. L. L. de Koninck. (*Chem. Zeit. Rep.*, 1905, xxix., 5.)—The process depends on the fact that, if the oxide contains metal or sulphide, these will reduce ferric salts to the ferrous condition. The ferrous salt formed will at once decolorize potassium permanganate solution, whereas the original metal or sulphide acts either not at all or only slightly on permanganate.

Two to 3 grammes of the zinc oxide are carefully rubbed down in a mortar with 20 to 30 c.c. water, in which has been dissolved 0·5 to 1·0 gramme of pure iron alum. 6 N sulphuric acid is then added gradually, with constant shaking, until the solution is complete. Excess must be carefully avoided. A single drop of permanganate solution should be sufficient to give a distinct rose colour. E. K. H.

Note on the Determination of Chromium in Steel. F. Ibbotson and R. Howden. (*Chem. News*, 1905, c., 3.)—The following method permits the estimation of the chromium to be determined on the portion of the sample taken for the manganese determination: To the liquid in which the permanganate has been titrated 50 c.c. of nitric acid (specific gravity 1·20) and 10 grammes of sodium bismuthate are added, and the mixture is heated slowly to boiling. As soon as a clear red solution is obtained, a little manganous sulphate is added to reduce the permanganic acid formed, and boiling is continued for one or two minutes. The small amount of manganese dioxide precipitated, which retains only a trace of the chromium, is filtered off, and the chromic acid determined in the filtrate, diluted if necessary, as usual with ferrous sulphate and potassium permanganate. A. G. L.

The Determination of Chromium in Steel. F. Ibbotson and R. Howden. (*Chem. News*, 1904, xc., 320.)—The method depends on the oxidation of the chromium to chromic acid in a nitric acid solution by means of ammonium persulphate in the presence of a small quantity of silver nitrate. Steels soluble in nitric acid (specific gravity 1·20) are dissolved in as small an amount of acid as possible. After expelling nitrous fumes and diluting copiously, 2 or 3 grammes of ammonium persulphate and 0·01 gramme of silver nitrate are added. The solution is heated until the evolution of gas commences, and allowed to stand for a few minutes until oxidation of the chromium and manganese is complete. If much manganese is present, it is precipitated as dioxide, which must be filtered off, but generally this does not take place. The solution is cooled, and the chromium precipitated as lead chromate by adding ammonium acetate and lead acetate. This precipitate is filtered off, washed, and dissolved in nitric acid, after which chromium is determined as usual. For the original oxidation the solution should not contain too much acid; if it is nearly neutral, the addition of silver nitrate is unnecessary.

For steels not soluble in nitric acid, 0·5 gramme of drillings is boiled briskly with 10 c.c. of sulphuric acid (1 : 4). When decomposition is nearly complete, 2 c.c. of nitric acid (specific gravity 1·41) are added, nitrous fumes are expelled, and the liquid is diluted up to 100 c.c. with warm water, any separated tungstic acid being disregarded; 20 c.c. of nitric acid (specific gravity 1·20) and 20 c.c. of 0·2 per cent. silver nitrate solution are then added, followed by 2 or 3 grammes of ammonium persulphate. The liquid is heated with vigorous shaking until the evolution of gas ceases, when it is boiled for a few seconds, cooled, and diluted. An excess of standard ferrous sulphate solution is then added, and the determination completed with dichromate solution. Excellent results are obtained unless manganese is present, any manganese being reckoned as chromium.

Steels containing large quantities of chromium and tungsten are decomposed with hydrofluoric and nitric acids, the solution being then evaporated with a little sulphuric acid and treated as above. An alloy containing over 30 per cent. of tungsten and 11·34 per cent. of chromium yielded 11·35 per cent. by this method, 0·25 gramme of the sample being used. A. G. L.

Colorimetric Determination of Phosphorus in Iron and Steel. T. E. Hewitt. (*Proc. Engineers' Society of W. Pennsylvania,* xx., 1904, 432.)—The colour obtained on passing hydrogen sulphide into an alkaline phosphomolybdate solution is employed for the colorimetric determination of phosphorus, and also of molybdenum. For the determination of phosphorus, 2 grammes of pig-iron or steel are dissolved in 40 c.c. of nitric acid (specific gravity, 1·20), and the solution evaporated to dryness. The residue is baked until all acid fumes are driven off, and, when cool, 30 c.c. of strong hydrochloric acid are added, and the mixture heated rapidly until insoluble basic ferric chloride first appears on the bottom of the vessel. Ten c.c. of strong nitric acid are next added, followed by 50 c.c. of cold water when the violent action has ceased. The solution is filtered and the siliceous residue on the paper washed with 2 per cent. nitric acid solution. To the filtrate strong ammonia is added until a permanent precipitate is produced, when strong nitric acid is added in slight excess. The solution, which should now have a clear amber colour, is heated to 85° C., and 50 c.c. of ammonium molybdate solution quickly added and the liquid thoroughly shaken for five minutes. (This molybdate solution is prepared by dissolving 225 grammes of molybdic acid in a mixture of 600 c.c. of water and 400 c.c. of strong ammonia, and adding the solution to 2½ litres of nitric acid of 1·20 specific gravity. The liquid is filtered after being kept in a warm place overnight.) When the precipitate has subsided, it is filtered, and washed with 2 per cent. nitric acid solution.

The yellow precipitate is now first "churned up" with very hot water, and a known amount of approximately decinormal caustic soda added until a clear solution results in the filter. When this solution has run through the filter into a 100-c.c. flask, the operation is repeated until all the precipitate is dissolved. Half of the total volume of caustic soda used is now added in excess to the solution of the precipitate, and the liquid made up to 100 c.c. A larger excess of alkali should be avoided. An aliquot portion of this solution is then diluted with water to about 30 c.c. in a Nessler tube, and hydrogen sulphide passed into the liquid for five minutes, when saturation should be complete. The tube is placed in boiling water for five minutes, and the contents diluted to 50 c.c. The yellow colour obtained is compared with a standard prepared in a similar manner. A standard phosphomolybdate solution of suitable strength is obtained by adding sufficient decinormal caustic soda to 0·2737 gramme of the yellow precipitate (prepared by precipitating sodium phosphate solution with the ammonium molybdate reagent, washing the precipitate with 2 per cent. nitric acid, and drying) to dissolve it, and then half the amount of caustic soda again in excess, and making the solution up to 500 c.c. with water. Ten c.c. of this solution contains 0·000009122 gramme of phosphorus. The colour tints are judged by looking through the column of liquid.

A number of test analyses are appended, showing that the method is capable of considerable accuracy. A. R. T.

The Gravimetric Estimation of Calcium. Utz. (*Oest. Chem. Zeit.*, 1904, vii., 510.)—The author finds that Brueck (*Zeits. f. angew. Chem.*, 1904, xvii., 945) is wrong in his statement that calcium cannot be quantitatively precipitated from an acetic acid solution, as well as from an ammoniacal solution. A series of experiments have shown that the character of the solution has no influence on the precipitation by means of ammonium oxalate.

An important point observed was that precipitation from an acid solution gave a more crystalline precipitate, thus facilitating filtration. H. A. T.

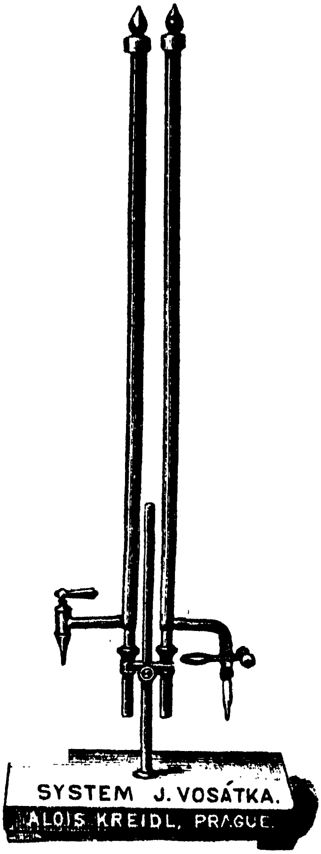
Methods for the Detection of Acetates, Cyanides, and Lithium. Stanley R. Benedict. (*Amer. Chem. Journ.*, xxxii., 480.)—*Acetates.*—The method depends on the facts that silver acetate is soluble in water and that the degree of ionization of acetic acid [illegible] reduced by the presence of an acetate. The solution is freed from all metals [illegible] those of the alkali group, and made just alkaline with sodium carbonate. [illegible] of silver nitrate is then added and the precipitate filtered off, leaving the solution perfectly neutral. The filtrate is next saturated with hydrogen sulphide and mixed with a solution prepared by adding two or three drops of normal acetic acid to 2 c.c. normal cobalt nitrate solution and saturating with hydrogen sulphide, any slight precipitate of cobalt sulphide formed being disregarded. If an acetate was present in the original solution, a heavy precipitate of cobalt sulphide will be produced when the two solutions are mixed. The test is capable of detecting acetate in $\frac{N}{500}$ solution; it is applicable in the presence of all strong acids and of all weak acids having insoluble silver salts.

Cyanides.—To test for cyanides in presence of thiocyanates and ferrocyanides, the solution is made alkaline with sodium hydroxide, and then about 0·5 c.c. of $\frac{N}{25}$ mercurous nitrate solution is allowed to flow slowly down the side of the test-tube, a ring of black mercurous oxide being thus produced. On gently shaking the tube, a portion of the precipitate will dissolve, while the remainder will become light gray in colour, owing to the formation of metallic mercury, if a cyanide is present. The test will detect cyanide in 5 c.c. of a solution containing 1 part CN in 1,000,000 parts of solution. For very delicate work a blank test may be made.

Lithium.—Advantage is taken of the slight solubility of lithium phosphate in hot or cold dilute alcohol, sodium phosphate, on the other hand, being difficultly soluble only in cold dilute alcohol. To the solution to be tested a little ammonia and one-tenth its volume of $\frac{N}{3}$ sodium phosphate solution are added, and then enough ethyl alcohol to produce a fairly heavy permanent precipitate. On heating the liquid to boiling, any lithium phosphate present will remain insoluble, whilst the sodium phosphate will dissolve; if the quantity of lithium present is very small, the solution will first become clear, and the lithium phosphate will only form after some boiling. Potassium does not interfere with the reaction. A. G. L.

Examination of Different Methods of Standardizing Potassium Permanganate Solution. H. Cantoni and M. Basadonna. (*Ann. de Chim. anal.*, 1904, ix., 365-371.)—Details of experiments are given from which the conclusion is drawn that, in general, the different methods employed for this purpose are not very exact. The easily-applied method of standardizing on piano-wire is not to be recommended, owing to the impossibility of obtaining wire of sufficient purity. Thus, four separate samples gave results differing by 0·00002393 gramme of active oxygen per c.c. of permanganate solution. The impurities, consisting of carbon, manganese, copper, etc., usually amount to about 0·45 per cent. Where very accurate work is not essential, the authors advocate taking a long piece of the wire, and cutting it into very small particles, so as to obtain a homogeneous sample, which should, however, be analysed by a gravimetric process. Where an absolutely exact result is required, the permanganate should be standardized on pure iron obtained electrolytically. The oxalic acid method yields concordant results, but these are generally too high, owing to the adventitious water that the crystals can retain. The use of anhydrous oxalic acid, recommended by Hampe, is not advantageous, as it is very hygroscopic and difficult to weigh. Ferrous ammonium sulphate gives more discordant results than either piano-wire or oxalic acid, this being due to the difficulty of obtaining the salt perfectly free from the isomorphous sulphates of potassium, magnesium, etc. Volhard's method of running the potassium permanganate solution into a mixture of potassium iodide and pure hydrochloric acid, and titrating the liberated iodine with standard thiosulphate solution, is that which gives results in closest agreement with standardization on electrolytic iron, and is the one recommended by the authors where the electrolytic process cannot be used. C. A. M.

APPARATUS.



Burette and Stand. Vosátka. (*Chem. Zeit.*, 1904, lxviii., 795.)—The burette is furnished with a solid continuation or rod, fitting firmly in a metal socket. This socket slides on the vertical support, can be clamped in any position, and, while securely holding the burette, does not obscure or cover the graduated scale.

Made by Alois Kreidl, Prague. H. A. T.

Volatilization of Platinum. G. A. Hulett and H. W. Berger. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1512.) — The authors show that platinum loses in weight when ignited in air or oxygen. The phenomenon appears to be due to the formation of volatile oxygen compounds of the platinum metals, stable at high temperatures, but decomposed at lower temperatures, and takes place with the very purest platinum as well as with the ordinary metal used for making crucibles, etc. The lowest temperature at which the effect is noticeable is 800° C.; at 900° C. the loss in weight may amount to 0·25 milligramme per hour per 100 square centimetres of surface; at 1,000° C. to 1 milligramme. The effect is not negligible for exact analytical work, since direct measurements showed that a good Bunsen burner heats a platinum crucible to about 960° C., and a blowpipe flame to about 200° higher. In a reducing atmosphere, or *in vacuo*, however, no volatilization occurs even at the highest temperatures of an electrically-heated muffle. A. G. L.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

The Twenty-seventh Annual General Meeting of the Institute of Chemistry was held at 30, Bloomsbury Square, on Wednesday, March 1, Mr. David Howard, President, in the chair.

The accounts for 1904 having been submitted by Mr. A. Gordon Salamon, Hon. Treasurer, and duly received, Professor Frank Clowes moved the adoption of the annual report of the Council, at the same time commenting on the progress of the Institute. He referred to the increase of 38 members during the past year. The register now contains the names of 973 Fellows and 163 Associates, making in all 1,136 members. The number of candidates for the examinations had increased to such an extent that it was found necessary to hold both intermediate and final examinations three times a year. After commenting on other matters in the report, Professor Clowes remarked on the growth of the library. Concluding, he said that the Institute was doing excellent work, and its qualifications and examinations were receiving more and more recognition. He was of the opinion that the time had come when the diploma of F.I.C. or A.I.C. should be insisted on, as evidence of competency, in connection with professional chemical appointments.

Mr. H. J. HELM, I.S.O., formally seconded the report.

Dr. G. T. MOODY said that he was disappointed to find no mention in the report of the question as to the subjects of preliminary examination. He alluded particularly to the retention of Latin as a compulsory subject.

The PRESIDENT said that the question had been under the consideration of the Council, and he felt sure that it would be raised again.

The report was then formally received and adopted.

Sir Thomas Stevenson, Professor J. Millar Thomson, Professor William A. Tilden, and Dr. John A. Voelcker were elected Censors; and Messrs. C. H. Cribb, R. E. Alison, and W. T. Burgess were appointed Hon. Auditors.

The PRESIDENT then delivered his address. Before commenting on the matters which had engaged the attention of the Council during the past year, he referred to the loss the Institute had sustained by the death of several distinguished Fellows. He specially mentioned Mr. Alfred H. Allen, an earnest worker for applied chemistry, who had taken an active interest in the Institute since its foundation, and Mr. William Chattaway, who had for several years assisted in the examination work of the Institute. He remarked on the steady growth of the Institute, saying that he thought there was still a wide field for those possessing the highest chemical knowledge and skill, and that those who had to call in the aid of such knowledge and skill were becoming more and more alive to the importance of employing only the properly trained and competent. He was of opinion that the training prescribed by the Institute and the high standard of the examinations had resulted in a decided improvement in the status of professional chemists. He emphasized the importance of requiring all candidates to produce evidence of a high standard of general education. The professional chemist should be a *professional* man as well as a chemist, and must, therefore, possess that general culture which is essential if he is to deal with his work in a professional spirit. He warmly supported the view, hitherto held by the Council, that Latin should be retained as a compulsory subject in the preliminary examinations.

Referring to the position of the Institute in connection with the Sale of Food and Drugs Acts, he mentioned that 94 per cent. of the Public Analytical appointments were held by Fellows of the Institute. He drew the attention of the members to the memorial presented by the Association of Public Analysts of Scotland to the Local Government Board (Edinburgh). They had been obliged to draw the attention of the Board to the fact that a number of authorities in Scotland were in the habit of submitting test samples to persons whose qualifications had not been approved by the Board, which practice was not calculated to secure the proper administration of the Acts.

The Government of India and the India Office had considered communications made to them by the Institute with reference to the practice of professional chemistry in India. The President drew the attention of the Fellows to a resolution recently passed by the Government, in which the qualifications F.I.C. and A.I.C. were formally recognised for public analytical appointments in India. The Council had undertaken to consider under what conditions officers of the Indian Medical Service could be admitted to the examinations of the Institute.

The President then proceeded to deal with other matters mentioned in the report, and alluded to the action of the Board of Agriculture in encouraging provincial technical and agricultural colleges to undertake professional chemical work gratuitously, or at purely nominal fees.

In conclusion, he reminded the members that if they might congratulate themselves on the present position of the Institute, they should not lose sight of the fact that much of its success was due to those who had worked for it in its early history. They had realized that, much as chemists had done for the world in the past, still greater things must be done by them in the future, and an organization such as the Institute should tend to draw them together to work for the common good.

On the motion of Professor J. MILLAR THOMSON, seconded by Dr. JOHN A. VOELCKER, a cordial vote of thanks was accorded to the President for his address.

The officers and members of Council for the ensuing year were duly elected as follows:

President: David Howard.

Vice-Presidents: Edward John Bevan; Edward Divers, M.D., D.Sc., F.R.S.; Percy Faraday Frankland, LL.D., Ph.D., F.R.S.; Edmund Albert Letts, D.Sc.; Edmund James Mills, D.Sc., F.R.S.; John Millar Thomson, LL.D., F.R.S.

Hon. Treasurer: Alfred Gordon Salamon, A.R.S.M.

Members of Council: Bertram Blount; Charles Edward Cassal, Lieut.-Colonel; Alfred Chaston Chapman; Arthur Crozier Claudet, A.R.S.M.; John Norman Collie, Ph.D., F.R.S.; James Kear Colwell; James Johnstone Dobbie, M.A., D.Sc.; Bernard Dyer, D.Sc.; Martin Onslow Forster, D.Sc., Ph.D.; Richard John Friswell; William Gowland, A.R.S.M.; Arthur George Green; Oscar Guttmann; Henry James Helm, I.S.O.; James Hendrick, B.Sc.; Egbert Grant Hooper; Herbert Jackson; William Walker James Nicol, M.A., D.Sc.; William Jackson Pope, F.R.S.; Alexander Scott, M.A., D.Sc., F.R.S.; William Ashwell Shenstone, F.R.S.; Alfred Smetham; Arthur Smithells, B.Sc., F.R.S.; David Alexander Sutherland; Edward William Voelcker, A.R.S.M.; William Palmer Wynne, D.Sc., F.R.S.; Sydney Young, D.Sc., F.R.S.

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REVIEWS.

THE CHEMICAL SYNTHESIS OF VITAL PRODUCTS AND THE INTER-RELATIONS BETWEEN ORGANIC COMPOUNDS. By RAPHAEL MELDOLA, F.R.S., V.P.C.S., F.I.C., etc. (Vol. I. London: Edward Arnold. 1904. Price 21s. net.)

All students of chemistry owe a debt of gratitude to Professor Meldola for having undertaken the compilation of this very interesting and suggestive contribution to chemical literature. The work under review deals not only with those "vital products" which are actually formed in the organism as the result of cell activity, and which may therefore be said to be of truly "vital" origin, but includes all those "atomic complexes" into which such vital products are capable of being resolved (assuming, of course, that these complexes have been prepared synthetically in the laboratory), as well as all substances isolated from animals or plants, even when there is reason to believe that they have not existed as such in the organism, but have been formed as the result of secondary changes occurring during the process of extraction. The reasons given for thus extending the scope of the work are of a weighty character, and are fully set forth in the preface. In the introductory chapters the author deals at some length with the question of the existence of a special *vital force.* He says, "The testimony of pure chemistry cannot, *as it at present stands,* be legitimately interpreted into a direct negation of vitalism in any form. This negation may, and probably will, be made possible in the future when our chemical methods have been made to approximate more closely to the vital methods," and goes on to remark: "When we can transform sugar into alcohol in the laboratory at ordinary temperatures by the action of a synthesized nitrogenous organic compound, when we can convert glucose into citric acid in the same way that *Citromyces* can effect this transformation; when we can build up heptane, or cymene, or styrene, or when we can produce the naphthalene or anthracene complex in the laboratory by the interaction of organic compounds at ordinary temperatures, then may the chemist proclaim with confidence that there is no longer any mystery in vital chemistry." Whilst a consideration of the vast mass of accumulated facts contained in the body of this work may well engender pride in our marvellous achievements in the domain of synthetical chemistry, the author seeks to guard us against undue conceit by reminding us of the vast number of transformations which are constantly being carried out in Nature's laboratory, and which we are at present apparently as far as ever from reproducing artificially, and reminds us that the science of biochemistry is yet to come. In two prefatory chapters the author discusses organic chemistry and chemical synthesis from the biocentric standpoint, and explains the reasons which have induced him to treat his subject-matter throughout on biochemical lines. He says, "The ordinary chemical equation representing the genetic relationship of one vital compound to another is apt to delude those who are not experts in chemistry into the belief that it is all-sufficient, and that it 'explains' the biochemical process; as a matter of fact *the sign connecting the two sides of the equation stands for the whole unexplored region of biochemical transmutation.*" The above will sufficiently indicate the position taken by Professor Meldola in relation to his subject as set forth in the highly interesting chapters which serve as a preface to this work. The text embraces a very large number of naturally occurring compounds, and contains detailed information in connection with their natural sources and the various synthetical processes by which they have been prepared. Full references to the original papers are in all cases given, and the general arrangement (a matter of no little importance in a work of this description) is admirably clear. The volume is well printed, is provided with an excellent index, and appears to be remarkably free from typographical errors. Attention may perhaps be called to a few slips which the writer has observed, so that they may be corrected in future editions: *Holn* on p. 45 should obviously be *Holm*, *Ekenstein* and *Nonnose*, on the same page should of course be *Eckenstein* and *Nonose* respectively, and *Saatz* on pp. 245 and 247 is usually spelt *Saaz*. In paragraph "E" on p. 250 the letter "d" should prefix the word "fructose." The fact that the above trifling errors, in addition to the few mentioned in the list of errata and corrigenda, should be the only ones which the reviewer has been able to detect in the course of a somewhat careful perusal of the book is a testimony to the great care which must have been exercised in its production. The author might, perhaps, have distinguished somewhat more clearly between the designation *Saccharomyces* and *Schizosaccharomyces*. In some sections these expressions are used quite correctly, but in others it is not clear whether the letter "S" stands as an abbreviation for the one or the other. It is interesting to note that hydroquinone, which is specially referred to in one of the introductory chapters, has, since the publication of this work, been found to occur in considerable quantities in the leaf-buds of the pear-tree. The present volume deals with the literature of the subject to the end of the year 1902, and contains an appendix in which such additional syntheses are included as were accomplished while the main text was passing through the press. This work is one which cannot fail to be of the highest value, not only to the teacher and to the scientific investigator, but also to the chemical technologist, and the appearance of the second volume, which is promised by the author, will be looked forward to with the greatest interest. A. C. C.

CHEMISTRY IN DAILY LIFE. By Dr. LASSAR-COHN. Translated by M. M. PATTISON MUIR, M.A. Third Edition. (London: Grevel and Co. 1905.)

This the third edition of Pattison Muir's translation of Lassar-Cohn's famous lectures contains some few additions by the author, designed to render the book more perfect and to bring it up to date. These will further enhance the value of this well-known and deservedly esteemed popular scientific work. W. J. S.

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OBITUARY NOTICE.

PROFESSOR STEIN.

WE regret to learn that Professor V. Stein, of Copenhagen, died suddenly on February 1, at the age of fifty-nine years. He had a great reputation for work in technical and agricultural analysis, not only in Denmark, but also in Sweden and Germany. He will be best remembered by our readers from his collaboration in 1888 with the late Mr. A. H. Allen on the question of the low Reichert-Wollny figures given by some Danish butters, the results of which were published in the ANALYST, 1889, xiv., 5.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, April 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. E. R. Bolton, J. T. Dunn, D.Sc., E. K. Hanson, M.A., F. T. Harry, and F. R. Henley, M.A., were read for the second time; and certificates in favour of the following candidates were read for the first time: Gilbert John Alderton, B.Sc. (Lond.), A.I.C., The Nest, Eglinton Road, Plumstead, assistant to Mr. Bertram Blount; Oscar Guttmann, M.Inst.C.E., F.I.C., 12, Mark Lane, London, E.C., consulting chemist and consulting engineer; John Theodore Hewitt, M.A. (Cantab.), D.Sc. (Lond.), Ph.D. (Heidelberg), A.R.C.Sc., 8, Montpelier Road, Twickenham, consulting chemist, Professor of Chemistry in the East London Technical College; George Druce Lander, D.Sc. (St. Andrews), D.Sc. (Lond.), F.I.C., consulting chemist, Professor of Chemistry in the Royal Veterinary College, Camden Town, London, N.W.

Messrs. E. Hinks, B.Sc., and S. E. Melling, were elected members of the Society.

The following papers were read: "The Determination of Higher Alcohols in Spirits" (I.), by Philip Schidrowitz, Ph.D., and Frederick Kaye, A.R.C.Sc.; and "The Action of Slightly Alkaline Waters on Iron," by Cecil H. Cribb, B.Sc., and F. W. F. Arnaud.

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THE DETERMINATION OF OXYGEN IN COPPER.

By S. DICKSON.

(*Read at the Meeting, March* 1, 1905.)

The processes for the determination of oxygen in copper in use at the present time, and on which reliance can be placed, are two in number:

1. That devised by Mr. Bertram Blount.
2. That devised by Mr. Leonard Archbutt.

The first process is based on the complete fusion of the metal in a current of dry hydrogen, and the water formed collected in a U-tube containing pumice saturated with sulphuric acid.

The second process consists in heating the metal (in a fine state of division) to redness in hydrogen, and the loss in weight is taken as representing the amount of oxygen present. In considering the above processes, and assuming both methods of equal accuracy, the analyst would most probably select that which would be more easily conducted, and for which the apparatus necessary is to be found in everyday use in the laboratory. There is, however, one objection which limits the usefulness of the latter process, and that is, the pieces of metal must be of very small dimensions. The first process, on the other hand, has no such drawback; the size of the pieces is immaterial, and a lump may be and is used. The only objections are the high temperature required for the fusion of the copper, to attain which special apparatus is necessary, and, when using a blast furnace, the heating of the absorption apparatus by radiation, it being by no means easy to insulate it thoroughly. The somewhat lower results obtained by this method, when compared with those given by the process to be described, are, I think, due to such heating. The use of an electrically heated furnace, as described in the February number of this journal by the same author, would no doubt obviate the defect.

In considering the objections to the foregoing methods, and in an attempt to overcome the same, it occurred to me that the property of combination (or solution) of a high melting-point metal with one of low melting-point, at or about the fusing-point of the latter, would probably give the desired result—viz., complete fusion at a comparatively low temperature.

The following experiments were conducted to ascertain if oxygen could be determined accurately in this manner. The method adopted was as follows:

Ten grams of copper in one piece, and 20 grams of tin (previously fused in hydrogen), were placed in a boat and heated in a porcelain tube until complete fusion had taken place, dry hydrogen passing through the apparatus as in the fusion process. The water formed caught in the ordinary sulphuric acid and pumice U-tube.

Comparative determinations on the same samples were also made by the fusion method, and two by a *modified* Archbutt method, the gain in weight of the U-tube, and not the loss in weight of the metal, being taken as representing the oxygen present.

	Blast Furnace Method: Fusion Lump.	Heating Small Piece only.	Copper and Tin Lumps.
Sample I.	0·15	0·140	0·166
„ Ia.	prolonged heating.	0·166	—
„ II.	0·107	—	0·115
„ III.	0·163	0·174	0·188
„ IV.	0·132	—	0·146
„ V.	0·161	—	0·194
„ VI.	0·021	—	0·022

A blank experiment was made, using 10·9 grams of copper previously fused by the blast in hydrogen, and 24·1 grams of tin, also fused in hydrogen: the gain in weight of the U-tube amounted to 0·0001 gram.

To ascertain the limit of size permitted by the Archbutt method, an experiment was conducted on strips of copper, the dimensions of which were $\frac{1}{32}$ inch by $\frac{1}{16}$ inch by 1 inch. The metal was heated in a glass combustion-tube at as high a temperature as possible without undue softening of the glass.

The amount of oxygen found was 0·061 per cent. The heating was then continued for thirty minutes, but there was no increase in the weight of the U-tube.

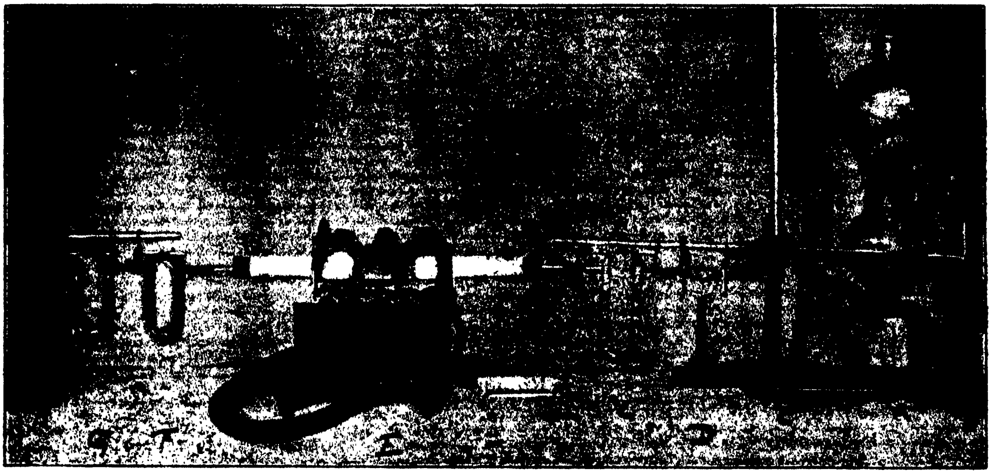
The original amount found was 0·115 per cent. (No. 11); therefore a deficit of 0·054 remains unaccounted for.

Twenty grammes of tin were then added to the metal used in the above experiment and the whole fused; the amount of oxygen remaining was:

Oxygen ... ... 0·050 per cent.

—or a total of 0·111 per cent., instead of 0·115 per cent. obtained by the tin solution method. A reasonably close agreement.

A further experiment was made on copper wire of 0·035 inch diameter. Ten grams were heated in the furnace to a red heat for two and a half hours, at as high a temperature as the glass tube would stand. The gain in weight amounted to 0·0075 gram. It was reheated for another period of one hour, and when weighed the increase amounted to 0·0008 gram. Again, after a further heating for one hour no increase occurred. The amount found by the copper-tin solution method being 0·0100 gram, or a deficit by the heating method of 0·0017 gram.



A, Hydrogen generator; B, wash-bottle; C, C, sulphuric acid and pumice drying-tubes; D, platinized asbestos heated with Bunsen burner; E, furnace with porcelain tube; F, weighed absorption-tube; G, guard-tube.

The apparatus consists of a hydrogen generator, drying-tube, platinized asbestos bulb tube heated by Bunsen burner for the elimination of oxygen, drying-tube, connected with the porcelain tube, 1⅛ inch external diameter, in which is placed the porcelain boat containing the copper and tin. The furnace consists of a section of Fletcher, Russell and Co.'s tube furnace, which is heated by a battery of five Bunsen burners. The absorption apparatus is a U-tube containing pumice saturated with sulphuric acid, and a similar one as a guard-tube. In conclusion, I have to thank Mr. R. H. Stanger, in whose laboratories the work has been carried out, and Mr. R. Doresa for the care he has taken in making the determinations.

DISCUSSION.

The **PRESIDENT** (Mr. Bevan) having invited discussion,

Mr. **ARCHBUTT** said that it was not altogether correct to speak of the "Archbutt method" for the determination of oxygen in copper. The method referred to was originally devised by Professor Hampe. He (Mr. Archbutt) had tested it, and, having found it to be a good method, had adopted it for his own use. He might claim also to have shown that it gave the whole of the oxygen without the trouble of fusing the copper, which was necessary in Mr. Blount's method. As he had mentioned in describing the method before the Society, comparisons of the two methods had been made, with the kind assistance of Mr. Blount; and with one exception, for which an explanation was given, the results had agreed very well. He was therefore not prepared to find that an increased quantity of oxygen was obtained as the result of adding tin to the copper. He did not understand why, in the experiment in which 0·061 per cent. of oxygen was obtained after one hour's simple heating of the copper in hydrogen, more was not obtained after heating for a further thirty minutes, if the copper contained more. The action certainly commenced on the outside, but he thought it gradually penetrated to the interior; and although he could quite understand that the oxygen might be given up slowly if the piece of copper were thick, he thought it would have been all given up eventually. He had not, however, experimented on the subject, and therefore could only express surprise at the result of Mr. Dickson's experiment. Since his paper was read he had used some specially-made silica glass bulbs, which could be heated to a very high temperature. He had found, however, that this was not really necessary, for practically the same results were obtained by heating to a good red heat in an ordinary Bohemian glass bulb. Sometimes a trifling amount more oxygen was obtained at the high temperature. Mr. Dickson seemed only to have noted the increase in the weight of the U-tube. He (Mr. Archbutt) took the loss of weight of the copper, and he thought it would have been more satisfactory if Mr. Dickson had done this as well as weighing the U-tube, because the one would have checked the other. The method which he used was, of course, more suitable for copper in very small turnings, which he could obtain without difficulty. If he had to deal with copper in large lumps, he should probably prefer such a method as Mr. Dickson's. His remarks had been called forth mainly by the discrepancies, which he should not have expected, in the analytical figures.

Mr. **BLOUNT** said that the author's process was certainly a very ingenious one. The only objection he could urge against it was that he did not like adding unnecessary materials in large quantity in any process of analysis.

Mr. J. H. B. **JENKINS** said that, in connection with the method used by Mr. Archbutt, the concave surface of the fine copper turnings was full of fissures and minute cracks, so that the surface actually exposed to the reducing action of the hydrogen was very great; and this fact should be borne in mind in comparing the behaviour of these small pieces of turnings with that of a solid block.

Mr. **DICKSON**, in reply, said that the whole question of the suitability of the different processes turned upon the state of division in which the metal was obtainable. Drillings or turnings were composed of very fine scrapings, agglomerated, perhaps, by the action of the tool, but still in a very fine state of division; and he quite agreed that, where it was possible to get the copper so finely divided, the whole of the oxygen could be estimated by a simple heating process. With a wire, however, it was difficult, if not impossible, to get the copper into such a fine state of division, and in that case a process of mere heating would fail. His reason for not weighing the copper was simply one of convenience, the experiments having been conducted in tubes. He had started with the idea that the heating method was quite a good one when the particles were fine, and had not considered it necessary to go any farther under those conditions. As to the effect of heating when the pieces of copper were larger, he showed a tube in which some pieces of copper wire had been heated to the highest temperature available, and, although the severity of the treatment was apparent, the attempt to obtain the whole of the oxygen in that way had distinctly failed.

Mr. Archbutt said that it would probably have been better had the heating not been so severe.

Mr. Dickson, however, thought that the higher the temperature the better the results would be.

The President said that the fact that Mr. Dickson had obtained a larger proportion of oxygen than either of the other processes yielded seemed to be in his favour, as he had shown that practically nothing was obtained in the blank experiment.

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SOME CONDITIONS AFFECTING THE ETHER VALUE OF BRANDY.

By PHILIP SCHIDROWITZ, Ph.D., F.C.S., and FREDERICK KAYE, A.R.C.Sc.

(*Read at the Meeting, March* 1, 1905.)

Some little while ago we analysed a brandy which showed an ether value* of 98·8, the alcoholic strength of the sample was 54·2. This sample, we were informed, was unreduced, and had been drawn direct from bond. At the same time we examined a sample which was stated to be the same brandy from duty paid stock, and reduced in strength to 44·5. To our surprise, the ether value of the latter was found to be only 66·5. At first we were inclined to doubt whether the two samples actually represented the same brandy originally, but our subsequent investigations lead us to say that we believe this to have really been the case. We were further informed that the spirit had been "broken down" with New River main water, the object of using this instead of distilled water being, according to our informants, to avoid the slightly mawkish taste that distilled water occasionally imparts to brandy. The cellar operations, which consist substantially in pumping the spirit into a vat, reducing therein with water, with subsequent filtration through paper pulp, and finally bottling, were carried out in the presence of one of us, and samples were drawn at various stages in order to ascertain, if possible, whether any of the operations or vessels were responsible for the falling off of the ether value recorded.

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* Expressed in grams per 100 litres of absolute alcohol. In order to avoid needless repetition, we may say that throughout this paper secondary products will be expressed in those terms, and alcoholic strength in per cent. by volume.

As a result, we found that none of the operations or vessels—apart from the "breaking down" with New River tap water, and disregarding for the moment the condition of the bottles—had any material influence in this direction. An examination of the influence of the reducing medium, however, afforded evidence that this factor must be taken into serious consideration by shippers and merchants. In addition, we are satisfied that the condition, or, rather, quality, of the glass bottles employed in bottling is a question deserving the serious attention of those interested. We propose briefly to allude to the latter point first.

The bottles examined by us were of clear white glass, stated to be of excellent quality, and guaranteed to be well cleaned before delivery. Half a dozen of these bottles were filled by us with distilled water, and a few drops of phenolphthalein solution were then added to the contents of each. In two cases a very pronounced pink coloration was produced at the point of contact of the fluid with the glass, but on shaking up this disappeared. After standing, however, for a few days,* the two bottles were observed to have developed a distinct pink colour throughout the liquid. After ten days the contents of the various bottles were titrated with $\frac{N}{10}$ acid, using methyl orange as indicator. The two bottles affected required 1·2 c.c. each for neutralization, the others only 0·3 to 0·4 c.c. It is fairly obvious, therefore, that bottles which display the slightest alkaline reaction should be rejected.

With regard to the influence of the nature of the water used for reducing, we were able to obtain direct evidence that the ether value is seriously affected thereby. The results of the experiments were as follows :

Number of Sample.	Alcoholic Strength.	Alcoholic Strength of Original.	Water Used for Dilution.	Ether Value.	Ether Value of Original.	Time in Days.
1.	44·37	55·46	New River main	66·9	78·2	7
Same as 1.	45·40	do.	do.	67·0	do.	do.
3.	39·14	55·88	do.	87·4	100·6	12
Same as 3.	39·09	do.	distilled	98·4	do.	do.
5.	33·15	55·60	New River main	55·2	69·3	2
Same as 5.	33·12	do.	distilled	62·7	do.	do.
7.	24·13	48·27	New River main	51·7	57·3	4
Same as 7.	23·62	do.	distilled	57·3	do.	do.

N.B.—The *time* refers to the number of days the mixtures of brandy and water were allowed to stand before being analysed.

From the above it will be seen that distilled water exercises very little, if any, influence on the ether value, but that an undistilled water (of otherwise excellent quality) may cause a falling off of nearly 15 per cent., if not more. The figures given at the commencement of this paper indicate that a loss of as much as 30 per cent· may arise from this cause.

We found also, as might have been expected, that the use of New River tap water appreciably affected the acid value of brandies. The following results illustrate the differences obtained in this regard, using distilled and tap water respectively as diluents.

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* We need scarcely say that all the bottles were kept tightly corked and completely filled throughout.

No.	Water Used.	Total Acid.
3.	[Original brandy] ...	128·8
	Distilled water ...	125·0
	New River main water	80·6
4.	[Original brandy] ...	46·6
	Distilled water ...	58·6
	New River main water ...	32·5
5.	[Original brandy	43·7
	Distilled water	55·3
	New River main water ...	13·3

We further observed that the use of tap water influenced the colour of the spirit to a much greater extent than does distilled water.

Having obtained these results, it seemed to us to be of interest to ascertain whether any other conditions, more particularly the blending of different brandies, materially affected the ether or other values. With this end in view, we made a number of blends, consisting of two brandies, each of which had been previously analysed. After blending, the samples were analysed at once, and in a number of cases were re-examined at the end of three weeks. For the sake of convenience we think it best to give the results for the total acid, non-volatile acid, and ethers in separate tables.

TOTAL ACID.

No. of Experiment.	Acid in Each Sample before Blending.	Theoretical Mean.	Found in Blend.	Increase or Decrease Per Cent.
1.	40·9 69·6	55·25	45·4	−17·8
2.	35·1 43·1	39·1	40·0	+ 2·3
3.	109·7 43·7	76·7	90·5	+15·2
4.	66·9 49·6	58·25	62·7	+ 7·0
5.	19·2 128·8	74·0	61·2	−17·2
6.	144·1 19·2	81·6	81·4	− 0·24
6*a**.	do.	do.	103·1	+20·8
7.	19·2 55·0	27·1	41·9	+35·3
8.	55·0 128·8	91·9	78·0	−15·1
8*a**.	do.	do.	89·6	− 2·3
9.	55·0 144·1	99·5	99·1	− 0·4
9*a**.	do.	do.	112·0	+11·1
10.	44·7 128·8	86·75	64·9	−25·1
10*a**.	do.	do.	66·0	−23·9
11.	44·7 62·4	53·55	50·0	− 6·6
11*a**.	do.	do.	52·7	− 1·5
12.	57·2 33·9	45·5	47·2	
12*a**.	do.	do.	47·2	+ 3·7

* Same sample after three weeks.

Looking at the above figures, it is clear that the total acid in a blend is by no means necessarily equivalent to the theoretical mean of the constituents. It also seems that time plays a not unimportant rôle in the changes that take place—that is to say, time quite apart from any actual maturing—as the above samples were kept in glass bottles full to the neck.

The deviation from the mean in the case of the non-volatile acid is not so marked as with the total acid, if we restrict this remark to the figures obtained immediately after blending, for in several cases very remarkable differences were observed after the lapse of three weeks—differences which we certainly cannot explain at present. Omitting the general table, which, as we have indicated, is of no particular interest, we subjoin figures in this connection which we believe to be worthy of attention:

NON-VOLATILE ACID.

No. of Experiment.	Acid in Each Sample before Blending.	Theoretical Mean.	Found in Blend.	Increase or Decrease Per Cent.
6.	63·0 4·2	33·6	36·7	+9·1
6*a**.	do.	do.	51·6	+34·8
8.	12·5 36·8	24·65	28·5	+15·6
8*a**.	do.	do.	44·7	+80·9
9.	12·5 63·0	37·7	33·7	−13·3
9*a**.	do.	do.	64·6	+71·3

With regard to the ethers, the deviations from the mean produced by blending were in a few cases found to be appreciable, and certainly larger than we have found the experimental error to be. At the same time, the results in this regard are not as striking as in the case of the acids. The following figures show the maximum deviations observed:

No. of Experiment.	Ethers in each Sample before Blending.	Theoretical Mean.	Found in Blend.	Increase or Decrease Per Cent.
3.	177·6 57·3	117·45	124·4	+ 5·8
8.	58·9 100·6	79·7	73·0	− 8·5
8*a**.	do.	do.	70·9	−11·0
11.	135·4 47·9	91·6	99·6	+ 8·7
11*a**.	do.	do.	93·5	+ 2·0
12.	47·0 106·3	76·65	82·5	+ 7·5
12*a**.	do.	do.	87·0	+13·5

* After three weeks.

Discussion.

The **President** (Mr. Bevan), referring to the alteration in acidity which accompanied the use of New River water, said that this would obviously be accounted for by the fact that that water was alkaline. It was remarkable, however, that the ethers also should have decreased on the addition of water, unless, indeed, the water contained a good deal of carbonate of soda. He had made one or two small experiments in this direction by mixing with alcohol small quantities of ethyl acetate and adding water containing in one case sodium bicarbonate and in another case calcium bicarbonate. Contrary, however, to the author's experience, he had not found any alteration to take place after several days. Obviously, a man might use any reasonable water-supply that he chose, and if in some cases the ether content was reduced, a very grave question was opened for the magisterial mind to consider.

Mr. **Fisher** said that he had recently had a sample of strong spirit from the Midi, which was said to be pure grape spirit, and which contained a high proportion of ethers, and it had occurred to him that if such a spirit were mixed with distilled water some of the ethers would hydrolyse, and that, in view of this possibility, it would be desirable to ascertain whether the acidity of the spirit was altered. Accordingly, he had diluted it with an equal volume of distilled water and redistilled the mixture, but had found that the acidity that came over was entirely unaffected; so that apparently there had been no hydrolysis that could be attributed to the addition of distilled water and subsequent distillation. That, in so far, was rather against the authors' conclusions.

Dr. J. T. **Hewitt** said that he was very glad this matter had been brought forward, because there were several things that must affect the ether value to a certain extent. For his own part, he was rather surprised that, on breaking down the brandy with a high ether value, so slight a diminution in the ether content had resulted. He should like to ask whether the authors had made any attempt to ascertain whether any constant value was obtainable for such an expression as—

$$\frac{\text{Number of molecules of ethers} \times \text{number of molecules of water}}{\text{Number of molecules of acid} \times \text{number of molecules of alcohol.}}$$

He had been wondering whether the authors were going to say anything about the flasks in which the estimations were made. This was really a very important point, and if thoroughly reliable flasks were not available, the only thing was to make a control determination, either beforehand or afterwards, with the same materials. He should also like to ask whether the author's ether estimations were made on the original samples or on the spirit after the removal of aldehydes. The latter mode of procedure was perhaps the more scientifically correct, but, as far as one could judge, most of the published results were obtained on the original samples, and any potash used in resinifying or effecting any other changes in the aldehydes had always been counted as ethers, so that for his own part he was rather inclined to take the total value obtained by working on the original sample. Moreover, it was difficult to effect the removal of the aldehydes satisfactorily, because nearly all the reagents available for the purpose were of such a nature that one could not quite say whether they would or would not have any effect on the ethers. One had to take into account the presence of hydrogen or hydroxyl ions which might affect the saponification and cause a low ether value to be obtained; and, again, certain reagents might act on the ethers and keep back part of their acids in the form of anilides or similarly constituted compounds.

Dr. Dyer suggested that these changes might conceivably be due to some biological influence, if the water were used straight from the tap and not filtered through a Pasteur filter.

Mr. Chapman said that Dr. and Mrs. Veley had some years ago described an organism which was capable of living and developing in tolerably strong rum, and it was not impossible that something of the same sort might occur, as Dr. Dyer had suggested, in the case of brandy. The question of the composition of the glass of the distilling flasks, which had been raised by Dr. Hewitt, was, however, a matter of much greater importance, and in his own laboratory he was in the habit of using carefully selected Jena glass for the purpose. The results which Dr. Schidrowitz had brought before them that evening were exceedingly interesting and curious, but he (Mr. Chapman) must confess that he would have liked to have seen a greater number of results with other kinds of water, such, for example, as the soft alkaline waters from the chalk underlying the London clay, and also with artificially prepared waters. Owing to its practical bearing, the statement made by Dr. Schidrowitz was a serious one, and it was obviously very desirable that further experiments should be made. He was rather surprised to hear that any spirit-merchant employed New River water for the purpose of breaking down brandy, as he should have thought that turbidity would have resulted.

Mr. Cribb said that he was somewhat inclined to question the possibility of these changes being entirely accounted for by the saponifying action of New River water. New River water contained about 18 parts per 100,000 of calcium carbonate, and, supposing a brandy to be diluted with half its volume of New River water, about a third of that amount, or 6 parts of calcium carbonate per 100,000, would be available for first neutralizing the acidity and then saponifying the esters. That would represent 12 parts of calcium carbonate per 100,000 parts of alcohol if the brandy contained 50 per cent. of absolute alcohol, and the quantity of acetic acid it could neutralize would only be a trifle greater. As the average amount of acidity was far greater than this, the possibility of the esters being attacked at all seemed to him to be very remote.

Mr. W. T. Burgess inquired whether the artificial waters used by the President were made up so that they contained what might be termed an excess of carbonic acid—for instance, by putting sodium carbonate or calcium carbonate into the water and passing carbon dioxide through it.

The President said that he had taken a saturated solution of sodium bicarbonate, estimated the soda, and diluted it down; and had then used a rather large quantity, representing about 70 parts of sodium carbonate per 100,000. In the case of the calcium carbonate he had simply passed an excess of carbon dioxide through water containing calcium carbonate in suspension. He did not, however, lay much stress on these experiments.

Dr. Dyer observed that one could not argue necessarily from ethyl acetate to the esters in brandy. The esters were calculated in terms of ethyl acetate, but that might under some circumstances be a very stable thing, whereas some esters might be very unstable.

The PRESIDENT said that it had merely occurred to him that, if there were any action, it would serve as confirmatory evidence. It did not, of course, follow that because there was no action on ethyl acetate there would be none in the case of actual brandy.

Dr. SCHIDROWITZ, in reply, said that, although it might be anticipated that the acidity should be diminished somewhat on the addition of New River water, it was remarkable that there should be such a diminution in mixed brandies. They had not as yet found any constant of the nature mentioned by Dr. Hewitt. He quite agreed with what had been said in reference in flasks. They were very careful as to this, and the experiments had been practically all made in the same flasks and under the same conditions. The aldehydes had not been removed previous to the estimation of the ethers. He had found in practice that the difference in the results due to the removal of aldehydes was, practically speaking, inappreciable. They had used the New River tap-water direct from the main, as it had been used in the case in question. With regard to Mr. Cribb's remarks, he could only say that the results of their experiments were purely matters of fact, and he might point out that they had not advanced the theory that the changes recorded were due to the alkalinity of the water. Possibly the explanation suggested by Dr. Dyer might be the correct one.

The PRESIDENT asked, as bearing on Dr. Dyer's suggestion, how long the blended brandies had been left to stand.

Dr. SCHIDROWITZ said that the mixed samples were examined immediately, and the same brandies were then left to stand for periods of three weeks, when the examination was repeated. In the experiments with water, the examinations were made after periods of two to twelve days had elapsed subsequent to the dilution.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

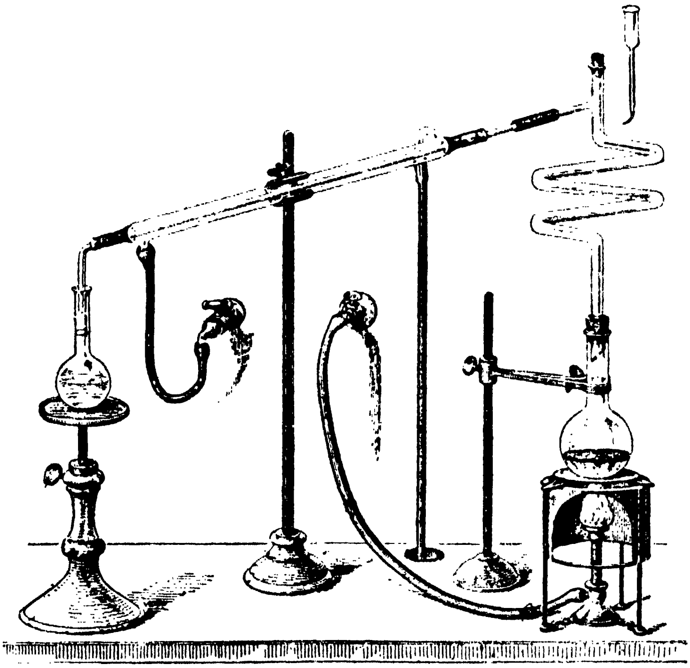
FOODS AND DRUGS ANALYSIS.

The Examination of Butters for Adulteration by Cocoanut Oil. A. Muentz and H. Coudon. (*Ann. d. l'Inst. Agron.*, 1904, iii., Part I.)—It is well known that the proportion of volatile soluble to the volatile insoluble fatty acids is much higher in butter than in cocoanut oil, and the authors have based on this difference a process for the approximate determination of the latter substance in butter. The method is as follows:

Ten grams of the fat, melted and weighed at 60° C., are weighed into a cylindrical vessel, and 5 c.c. of potash solution (120 grams KOH dissolved in water and made up to 100 c.c.) are added and stirred for ten minutes, when a hard soap is obtained. Saponification is then completed by heating to 70° to 80° C. for a further period of twenty minutes.

The saponified fat is washed into a distilling-flask with 200 c.c. of water and gently warmed until solution is complete. The fatty acids are then liberated by the addition of 30 c.c. of phosphoric acid (specific gravity 1·15), and any CO_2 given off is removed by connecting the flask to a pump for ten minutes.

It is important, in order to obtain comparative results, that the dimensions and general arrangement of the distilling apparatus should not vary. The flask, of Bohemian glass, has a capacity of 500 c.c., measured to the bottom of the neck. A fractionating tube is interposed between the flask and the ordinary condenser, and has a total length of 1 metre, with an internal diameter of 14 millimetres. To reduce the space required it is bent into a zigzag or serpentine form. This tube is connected to the condenser by a short lateral tube at the upper end, and its total available length from the top of the flask to the lateral tube is about 92 centimetres. The flask, resting upon a copper ring 6 centimetres in diameter, is heated by an ordinary Bunsen flame, regulated so as to complete the distillation in one and a half hours.



(One-tenth the actual size of the apparatus.)

Exactly 200 c.c. are distilled over, and allowed to stand until the following day before filtering through a wet paper. The flask is washed with water (5 c.c.), which is then poured through the filter, and the soluble fatty acids are titrated with lime-water, using phenolphthalein as indicator, the end-point being taken when a pink colour remains permanent for a few seconds. The results are expressed as butyric acid.

The filter is then washed with four lots of 5 c.c. each of alcohol, poured on drop by drop, the washings being collected in the flask originally used for receiving the distillate. Twenty c.c. of alcohol are introduced into the condenser, the bottom orifice of which has been previously closed; the alcohol should entirely fill the inner tube, and, after remaining for a few minutes, is run into the distillate flask, and the condenser rinsed with a further 5 c.c. of alcohol. All the insoluble fatty acids are now in the flask, and are titrated in this with lime-water, taking as end-point the formation of a permanent pink colour. The result is again expressed as butyric acid.

In forty analyses of pure butters of known origin the volatile insoluble acids averaged 0·65 per cent., and the ratio of these to the soluble fatty acids, according to the formula $\frac{\text{Insol. acids}}{\text{Sol. acids}} \times 100$, gave a value of 10 to 15. Cocoanut oil, on the other hand, gave a mean percentage of insoluble fatty acids of 3·405, or, according to the above formula, a value of 250 to 280.

The mean value for butter-fat was found to be 12·04, and it is suggested that this should be taken as the standard number.

Analyses of various mixtures of butter with cocoanut oil gave the following figures:

	$\frac{\text{Insoluble Acids}}{\text{Soluble Acids}} \times 100.$
Butter containing 50 per cent. cocoanut oil	73·1
,, ,, 20 ,, ,, ,, ...	27·0
,, ,, 15 ,, ,, ,, ...	24·1
,, ,, 12·5 ,, ,, ,, ...	22·9
,, ,, 10 ,, ,, ,, ...	19·8

The authors claim that by the employment of this method it is possible to detect with certainty as little as 5 per cent. of cocoanut oil in butter (*cf.* ANALYST, xxix., 154).

H. A. T.

The Analysis of Vioform and Vioform Gauze. W. Fresenius and L. Grünhut. (*Zeit. anal. Chem.*, 1905, xliv., 25-28.)—Vioform, which has recently been introduced as an antiseptic bandage dressing, is a bulky powder, with a composition corresponding to the formula $C_9H_4N.(OH)ClI$ (chloriodohydroxyquinoline). It dissolves readily in cold alcoholic potassium hydroxide, and the solution remains clear when diluted with a considerable quantity of water. The addition of nitric acid precipitates the vioform, and if the exact amount required to neutralize the alkali be added, the precipitation is quantitative. In an analysis the precipitate is collected on a weighed filter, which is then thoroughly washed with cold water, and dried at 100° C. until constant in weight. Test experiments showed that there was a loss averaging 2·62 per cent. during the drying.

Vioform Gauze.—From 6 to 8 grams of the sample are made into a roll and placed in a Soxhlet's extractor with sufficient $\frac{N}{2}$ potassium hydroxide solution to just cover it, whilst 75 c.c. of alcohol are introduced into the flask beneath. After standing for two hours, heat is applied to the flask, and the extraction with alcohol continued for several hours. The contents of the flask are then diluted with ten times the amount of water, and the vioform precipitated with nitric acid, dried, and weighed as above described. Finally a determination of the total amount of halogen is made, in order to prove the identity of the powder. Theoretically, vioform requires 70·69 per cent. of silver to combine with the halogens, whilst the author found 67·01 per cent. in a test experiment. C. A. M.

ORGANIC ANALYSIS.

The Determination of Alcohol by the Freezing-point Method. R. Gaunt. (*Zeit. anal. Chem.*, 1905, xliv., 106-108.)—The author's experiments have been made with the object of determing to what extent Raoult's method can be used as a practical means of determining alcohol in aqueous solutions. For this purpose Beckmann's apparatus was used, and the freezing mixture of ice-water and salt kept at about − 8° C. The following results were obtained:

Amount of Alcohol in 100 Grams of Solution.	Lowering of Freezing-point compared with that of Water. I.	II.	Calculated Decrease for each 1 per Cent. of Alcohol. I.	II.
Grammes.	° C.	° C.	° C.	° C.
1·0	0·428	0·420	0·428	0·420
2·0	0·853	0·845	0·426	0·422
3·0	1·271	1·267	0·424	0·422
4·0	1·692	1·690	0·423	0·422
5·0	2·120	2·135	0·424	0·427
6·0	2·554	2·570	0·425	0·428
7·0	3·010	3·020	0·430	0·431
8·0	3·510	3·520	0·439	0·440
10·0	4·525	4·530	0·452	0·453
12·0	5·590	5·600	0·466	0·467

Thus, up to 7 per cent. of alcohol the lowering of the freezing-point is proportional to the amount of alcohol, but at higher strengths it becomes greater. The error of determination corresponds to only about 0·25 per cent. of alcohol, which is negligible for most practical purposes. C. A. M.

A Source of Error in the Estimation of Acetone by the Iodoform Method. W. Vaubel and O. Scheuer. (*Zeits. f. angew. Chem.*, 1905, xviii., 214.)—The method of determining acetone as iodoform, due originally to G. Kraemer, was modified by J. Messinger, who treated the solution containing acetone with solution of potash (5 to 6 per cent.), then added excess of a standardized iodine solution, and, after two minutes' standing, estimated the excess of iodine in the usual way with thiosulphate.

Messinger's method, however, does not satisfy all requirements, and the authors have noticed for some time a source of error of especial importance, and the present communication is the outcome of their observations.

The principle involved has been worked out by Foerster and Gyr, whose work was not known to the authors at the commencement of their researches. A quotation from a reference to Foerster and Gyr's paper, "The Action of Iodine on Alkalies," in the *Chemisches Centralblatt* is given, of which the substance is as follows: When iodine is treated with alkalies, hypo-iodite is formed, but the reaction is incomplete, and free iodine exists concurrently with free alkali. The quantity of free iodine compared to a definite amount of hydroxyl ion is, in accordance with the requirements of the Law of Mass Action, being greater the more iodine ions the solution contains.

If bicarbonate is added to a solution of hypo-iodite, the quantity of free iodine is diminished, since the quantity of hydroxyl is decreased. There remains always a certain amount of hypo-iodous acid, as Foerster and Gyr deduce from the fact that the solution requires less thiosulphate than it does arsenious acid. They consider that free iodine converts thiosulphate into tetrathionate, whereas hypo-iodous acid oxidizes it directly to sulphate. Arsenious acid is not oxidized by free iodine, but only by hypo-iodous acid. Dissolved hypo-iodite gradually passes entirely into iodate. This takes place with very great velocity, but the more slowly the greater the excess of free alkali there is. Rise of temperature accelerates the reaction; increase in the amount of potassium iodide present also accelerates the formation of iodate. In consequence of the iodate being formed at the expense of the hypo-iodous acid, the quantity of free iodine (which is in equilibrium with the hypo-iodite) must also diminish.

The authors examined this action further, with the following result:

If 20 c.c. of $\frac{N}{10}$ NaOH solution and 20 c.c. of $\frac{N}{10}$ iodine solution were mixed, and then titrated back with thiosulphate, 4·0 c.c. $\frac{N}{10}$ thiosulphate were used up; and if acetic acid were then added, 13·2 c.c. more were required—in all, 17·2 c.c. Thus, there was still an amount of 2·8 c.c. of thiosulphate wanting, which should have been required if the iodine and alkali had reacted, to give one molecule of iodide and one molecule of hypoiodite.

Similar results were obtained with other mixtures by using thiosulphate, but when arsenious acid was used, after first acidifying and subsequent addition of bicarbonate, the correct result was obtained.

The conclusion of the authors is that, in order to avoid the error in Messinger's method, it is necessary to titrate back with arsenious acid, and not with thiosulphate.

The method suggested by Martz, of standardizing the thiosulphate solution by means of a known amount of iodine dissolved in sodium hydroxide, and again liberated by means of acid, does not completely effect the purpose, as the extent of the error varies with the proportion of iodine to alkali.

Further communications will follow. E. K. H.

A New Method of Distinguishing between Primary, Secondary, and Tertiary Alcohols. P. Sabatier and J. B. Senderens. (*Bull. Soc. Chim.*, 1905, xxxiii., 263, 264.)—The vapours of the alcohol are brought in contact with reduced copper heated to 300° C. and the products of the reaction examined. In the case of a primary alcohol, conversion into hydrogen and the corresponding aldehyde takes place. Secondary alcohols yield hydrogen and acetone, while tertiary alcohols are transformed into water and an ethylenic carbide (*carbure éthylénique*) which is usually liquid (gaseous in the case of trimethyl-carbinol). In making the test, the tube containing the reduced copper is closed at one end by a cork, through which passes a capillary tube communicating with an open tube containing the alcohol, whilst the other end is connected with a condensing vessel. The apparatus is first swept out by means of a current of hydrogen, the copper then brought to about 300° C., and the alcohol introduced through the capillary tube. The condensed liquids are examined for the respective products. If a coloration is given with Schiff's reagent (a solution of fuchsin exactly decolorized with sulphurous acid), the alcohol is *primary*. If no coloration is obtained, 1 gram of semicarbazide hydrochloride mixed with 1 gram of potassium acetate and 6 c.c. of water are added, and the formation of a precipitate immediately or after a few seconds indicates acetone and that the alcohol is *secondary*. The non-formation of a precipitate shows that the alcohol was tertiary, and the presence of an ethylenic carbide can be identified by the addition of a drop of bromine, which is immediately decolorized. C. A. M.

The Origin of Fusel Oils. O. Emmerling. (*Berichte*, 1904, xxxvii., 3535-3538.)—Under the ordinary conditions of alcoholic fermentation of pure worts containing different carbohydrates, only traces of fusel oil are produced by different kinds of yeasts. The author finds, however, that numerous anaerobic bacteria are capable of converting carbohydrates, notably starch and cane-sugar, into higher alcohols, especially propyl, butyl and amyl alcohols. Typical micro-organisms possessing this property are *Granulobacter butylicum* and *B. orthobutylicus*, and certain species of bacteria invariably present on the skins of potatoes. Fusel oil is only produced under anaerobic conditions, and much larger yields are obtained by the use of non-hydrolysed material, leaving the bacteria to effect the hydrolysis themselves. Hydrogen, carbon dioxide, butyric acid and traces of ethyl alcohol are also formed during the fermentation, which proceeds well at 37° C. The addition of nitrogenous compounds does not increase the yield of fusel oil. C. A. M.

A Direct Method of Estimating Glycerin. A. A. Shukoff and P. J. Schestakoff. (*Zeits. f. angew. Chem.*, 1905, xviii., 294.)—Most processes in present use in analytical practice for estimating glycerin depend on indirect methods, based on its oxidation products (Benedikt and Zsigmondy, Hehner), or saponification of its esters (Acetin method).

The difficulty in employing a direct method of extraction by shaking up an aqueous solution with any organic solvent lies partly in the limited solubility of glycerin in organic solvents, and partly in the great avidity with which it is retained by the water.

The authors have, however, succeeded in overcoming this difficulty, and in working out a practical method for the direct estimation of glycerin.

Their method consists in mixing the glycerin-containing solution with powdered anhydrous sodium sulphate, and extracting the mass so obtained with dry acetone. The process, in detail, is as follows:

If the solution to be analysed is alkaline, it is first made slightly acid with sulphuric acid, and any precipitate filtered off. The filtrate is then made slightly alkaline with potash; if the solution is originally acid, the potash can be added directly.

The solution so obtained is then concentrated to a syrup at a temperature not exceeding 80° (to prevent loss of glycerin); solutions which precipitate salts on concentration are evaporated to a semi-solid consistency.

A quantity of material should be taken which will yield not more than 1 gram of pure glycerin. Thus there is obtained a nearly dry powdery mass on mixing the evaporated liquid with 20 grams of ignited powdered sodium sulphate, which can be easily transferred to a paper thimble for the Soxhlet extraction apparatus. The form of Soxhlet used must be such that all joints are of glass ground to fit, as acetone attacks cork and rubber. The acetone used for extraction should be well dried over ignited potassium carbonate, and distilled. The extraction takes about four hours.

If, after distilling off the acetone, the glycerin shows oil-drops on the surface, these can be got rid of by washing with low-boiling petroleum spirit.

The glycerin is now dried in an air-bath at 75° to 80°, till the weight is constant (usual time four to five hours). The temperature must not exceed this limit, and the bulb of the thermometer should be placed close to the side of the flask, which is closed by a well-fitting stopper for weighing.

Solutions containing more than 40 per cent. of glycerin do not need concentration, but can be treated with sodium sulphate at once.

The authors give figures which show comparative results by three methods: direct extraction as above, specific gravity, and Hehner's method. The figures indicate that the new method is quite equal in accuracy to the old. E. K. H.

Detection of Dextrose by Barfoed's Cupric Acetate Method. H. C. Sherman. (*American School of Mines Quarterly*, 1905, xxvi., 159, 160.)—This method* is useful for distinguishing between dextrose (or other monosaccharide) and those disaccharides which also reduce Fehling's solution. The solution employed is prepared by dissolving 45 grams of neutral crystallized cupric acetate in 900 c.c. of water, filtering if necessary, adding 1·2 c.c. of 50 per cent. acetic acid, and diluting to 1 litre. A portion of this solution when heated in a water-bath must show no change.

Five c.c. of the solution to be tested are mixed in a test-tube with 5 c.c. of the reagent, and heated in a water-bath for three and a half minutes. The contents of the tube are then examined for cuprous oxide, viewing the tube against a black background in a good light. If no evidence of reduction be found the tube is kept at the ordinary room temperature for ten minutes and again examined.

Under these conditions maltose and lactose do not reduce the reagent, but longer heating or the use of a more acid solution causes a reduction, owing to the hydrolysis of the disaccharide. In order to prevent this, the solution may be heated to 40° C. only. At this temperature, however, dextrose itself reduces so slowly that the heating must be continued for several hours. The test is capable of distinguishing between dextrose and maltose in aqueous solutions containing less than 0·02 per cent. of the former and 0·2 per cent. of the latter, provided that the results are controlled by check experiments with solutions of known amounts of dextrose and maltose. Dextrin does not interfere, but if proteids are present in the solution to be tested they are precipitated by the copper, and render the test slightly less delicate. W. P. S.

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* This old process is given because exact directions for working it are now so rarely to be found.

Determination of Sugar in Urine. J. Bilinski. (*Monatsheft Chem.*, 1905, xxvi., 133-141.)—Varying quantities of the urine are heated in a series of test-tubes, each tube containing 6 c.c. of Fehling's solution. To determine in which tube the volume of urine added has exactly reduced the Fehling's solution, a few drops of uranium nitrate solution are added, and the tubes again heated. In those tubes containing the slightest excess of sugar above the quantity necessary to reduce Fehling's solution, the uranium nitrate is also reduced, and a greenish or brownish coloration is produced. The details of the process are as follows: 50 c.c. of the urine are treated with so much 4 per cent. uranium nitrate solution that a drop of the mixture gives a brown-red colour with a little powdered potassium ferrocyanide. The solution is then diluted to 100 c.c. and filtered. A series of preliminary tests are now made to ascertain how much of this solution is approximately required to reduce 6 c.c. of Fehling's solution. According to the quantity of sugar thus found, the urine solution is diluted to five, seven, or ten times its volume, and quantities of it differing from each other by 0·1 c.c. and about equal to the volume required to effect complete reduction are mixed in test-tubes with 6 c.c. of Fehling's solution and a few drops of uranium nitrate solution. The tubes are then heated almost to boiling and allowed to settle. Should one tube exhibit a yellow coloration and the next one, containing 0·1 c.c. more urine solution, a green colour, the first contains the exact amount of sugar (in the urine) to reduce the 6 c.c. of Fehling's solution—that is, the volume of urine in this tube contains 0·03 gram of sugar.

The method works well with urines containing more than 0·6 per cent. of sugar. With smaller quantities, a little solid sodium hydroxide must be added to each tube, as, with the necessarily large volumes of urine added, the dilute alkaline solution fails to give a satisfactory coloration. Exactly known weights of grape sugar may also be added to urines containing little sugar, in order to obtain a satisfactory coloration, the amount of added sugar being afterwards subtracted from the quantity found.

W. P. S.

A New Method of Esterifying Organic Acids. A. Werner and W. Seybold. (*Berichte*, 1904, xxxvii., 3658-3661.)—This is based upon the fact that dimethyl sulphate reacts upon the alkali salts of organic acids to form the methyl ester of the acid in question. Thus—

$$R.\mathrm{COOK} + (\mathrm{CH_3})_2\mathrm{SO_4} = R.\mathrm{COOCH_3} + \mathrm{CH_3KSO_4}.$$

Five grams of the acid are dissolved in a slight excess of N-potassium hydroxide solution, and the liquid well shaken for thirty minutes with twice the molecular proportion of dimethyl sulphate. It is next heated for thirty minutes on the water-bath to destroy the excess of the reagent, after which it is cooled and an excess of potassium hydroxide added to separate the ester from the free acid. Experimental results are given to show that satisfactory yields are obtained by this method with acetic, iso-valeric, stearic acids, etc. The method can also be used for the methylation of acids, such as 2.4.6 tribromo-benzoic acid and 2.4.6 trinitro-benzoic acid, where the usual method of esterification is impracticable.

C. A. M.

The Determination of Sulphuric Acid in Malt Worts and the Ash of Beer. W. Windisch. (*Wochens. Brauerei*, 1905, xxii., 17; through *Chem. Zeit.*

Rep., 1905, xxix., 49.)—The author calls attention to the well-known fact that the amount of sulphuric acid found in the ash of malt worts or beer is much lower than that actually present, unless the ignition took place in the presence of a base as sodium or barium hydrate.

The loss of sulphur by reduction is considerably greater than has hitherto been assumed. A series of analyses are quoted, showing that if an alkali is used in the ignition the amount of sulphuric acid found is very materially higher than would ordinarily be the case when no alkali had been employed. H. A. T.

Determination of the Viscosity of Lubricating Oils. R. Hackel. (*Mitt. kaiserl. königl. tech. Gewerbe-Museum in Wien*, 1905, xv., 44-51.)—Experimental determinations are given showing that determinations made with a ring burner in Engler's vicosimeter involve smaller possible errors at 50° C. and 100° C. than those made without such a burner. The difficulties in the latter case are obviated in an apparatus devised by the author, in which the viscosimeter is connected with two other vessels containing oil. One of these is heated by means of a Bunsen burner, while the oil in the other is cooled by a cold-water coil, so that the contents of the viscosimeter can be brought to any required temperature by the admission of either hot or cold oil to the heating chamber, which is ring-shaped, and is provided with an additional outlet-tap for drawing off oil that has been made too hot or too cold. C. A. M.

The Determination of the Solidification-point of Lubricating Oils. R. Hackel. (*Mitt. kaiserl. königl. tech. Gewerbe Museum in Wien*, 1905, xv., 38-43.) —The rapid method of determining the solidification-point of oils by immersing the tube in a mixture of ice and salt is shown to involve errors due to too rapid cooling, and the amount of these is too variable to admit of the use of any factor of correction. The most exact method is to keep the tube of oil immersed for at least an hour in successive freezing mixtures, consisting of ice and water with the following salts in solution, and contained in a vessel of at least 100 mm. in diameter:

To Produce an Approximate Temperature of— °C.	Salt.	Parts by Weight in 100 Parts of Water.
− 3	Potassium nitrate	13·0
− 5	Potassium nitrate and sodium chloride	13·0 and 3·3
− 9	Barium chloride	35·8
− 10	Potassium chloride	22·5
− 15	Ammonium chloride	25·0
− 18	Sodium nitrate	50·0
− 21	Sodium chloride	33·0

For more rapid determinations the author has devised an apparatus in which the oil is cooled by ice and salt much more slowly than in the ordinary method, so that the results agree more nearly with those obtained by use of the above solutions. In this apparatus the test-tube of oil is fixed in a larger tube, and both are clamped in a vessel with an exterior chamber containing the ice and salt; so that there is a double intervening air-spare between the oil and freezing mixture. The tube is closed by a rubber cork through which a thermometer passes, and an observation-slit in the outer vessel enables the behaviour of the oil to be watched, whilst the tubes can be raised and shaken in the clamp without coming in contact with the hand. C. A. M.

The Determination of Rosin in Shellac. A. C. Langmuir. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 12.)—The petroleum-ether extract of shellacs gives no reliable quantitative indication of the rosin present, while the test based on the formation of a green colour on shaking the petroleum-ether extract with dilute copper acetate solution fails to show a less proportion than 15 per cent. of rosin. The well-known Storch-Morawski reaction may be made to detect as little as 2 to 3 per cent. of rosin in shellac by treating 1 gram of the sample with 15 c.c. of acetic anhydride, and warming the mixture until solution is complete. On cooling the liquid the rosin remains in solution, and may be filtered from the gelatinous shellac. Two drops of strong sulphuric acid are then allowed to flow down the side of the tube containing the acetic anhydride solution of the rosin, and the liquid gently shaken. An evanescent coloration is produced in the presence of even 2 per cent. of rosin. Pure shellacs give no colour if charring has been avoided.

According to the author, the Wijs' method of determining the iodine absorption is the best process for the examination of shellac, since it provides both a qualitative and quantitative test for rosin. Shellac has no effect on the colour of the Wijs' iodine chloride solution except after long standing; while rosin, even when mixed with shellac, gives a characteristic red-brown colour in proportion to its amount, and by means of standards a rough colorimetric determination may be made.

The well-known difference between the iodine values of rosin (high) and shellac (low) has caused a determination of the iodine absorption to be considered the most valuable as a test of purity of the latter. The author prefers Wijs' modification, which he works as follows: 0·2 gram of ground shellac is treated with 20 c.c. of glacial acetic acid in a 250 c.c. stoppered bottle, and the liquid warmed somewhat until solution takes place (the wax remains insoluble). Solution takes place more quickly in proportion to the amount of rosin present, shellac being only difficultly soluble. Ten c.c. of chloroform are added, the solution cooled to 21° to 24° C., and kept at that temperature during the determination. Twenty c.c. of the Wijs' solution are next run in from a pipette having a fine orifice, and the stoppered bottle then laid aside in the dark for exactly one hour. The process is then carried out in the usual way. In the case of bleached shellac, 0·4 gram is used in the determination, while for rosin 0·15 gram of sample and 40 c.c. of Wijs' solution are the best quantities. The temperature and time of reaction should be constant, since the author has found very large differences in the results obtained when variations in these conditions are introduced. A liberal excess of Wijs' solution should be added. The effect of the shellac-wax (iodine value 4·5) is small, and may be ignored.

The author regards 18 as the maximum iodine value (Wijs') of a pure shellac examined under the above conditions, and the value 228 as an average for the very variable rosin figures. Shellacs giving 18 to 23 iodine absorption may be regarded as "fair"; at 23 the various qualitative tests show the presence of rosin, while samples giving 23 to 28 are "poor."

A close connection is traceable between the iodine absorption of a sample of shellac and the quantity of bleaching agent required to bleach it properly. Rosin is bleached only with difficulty and by the addition of a much larger quantity of sodium hypochlorite solution. The colour of the "varnish" obtained by dissolving the bleached and washed shellac, separated from its solution by acid in alcohol in the proportion of 4 pounds shellac to 1 gallon of the spirit, should be compared with that obtained from a sample of known purity. Bleached shellacs have a lower iodine value than unbleached samples, the average figure being about 8 per cent. A figure much exceeding 10 points to adulteration, and for bleached shellacs the figures used in the formula for calculating the proportion of rosin should be 10 and 228.

Much adulteration is practised in the preparation of shellac-varnish. The strength or "body" of the varnish may be determined by weighing the solid matter obtained on evaporation of the solvent, while an examination of the latter will show its character (wood-spirit, grain-spirit, petroleum spirit, etc.). Rosin may be detected by the iodine value of the solid residue, which should be about 10 to 15. In the case of a large proportion of rosin, the iodine value of the residue will scarcely give a reliable figure, since the iodine absorption of rosin is materially lowered on heating. It should also be noted that other alcohol-soluble resins, such as sandarac, copal, and kauri, may be present as adulterants in shellac-varnish. The following is a simple and reliable colorimetric test for the presence of adulterants in shellac-varnish; and the test may be readily applied to shellac by dissolving it in alcohol. Five c.c. of glacial acetic acid and 5 c.c. of Wijs' solution are added to 5 drops of the varnish. With pure shellac-varnish the liquid will remain light yellow with a shade of red, while an impure varnish will almost immediately develop a reddish-brown colour, proportional to the rosin, etc., present. Sandarac, copal, kauri, and acaroid resins act like rosin in this test, while their iodine values (Wijs) range from about 120 to 175. A. R. T.

Note by Abstractor.—E. J. Parry, in the *Chemist and Druggist*, April 7, 1905, has replied to some of the criticisms in Langmuir's paper, and pointed out certain advantages of the Hübl method over the Wijs' modification of the iodine absorption in the case of shellacs.

The Gum of Feronia Elephantum. P. Lemeland. (*Journ. Pharm. Chim.*, 1905, xxi., 289-295.)—The *Feronia elephantum*, which belongs to the family of *Auriantiaciæ*, is indigenous to the East Indies, but is also cultivated for the sake of its fruit and gum in Ceylon and Java. The gum is sold in the bazaars of Madras under various titles, such as *wood-apple gum*, *katbêl-ki-gond*, *velampishin*, *kapithamia piscum*, etc. It is also sold in England, where it is stated to be frequently adulterated with gum arabic. The sample examined by the author was in the form of small brilliant translucent particles of about the size of a small pea, some colourless, and others yellow or reddish-amber. It contained 17·75 per cent. of moisture, and 6·9 per cent. of substances insoluble in water. The optical rotation of the soluble portion at 15·5° C. was $[\alpha_D] = -6°·41'$. It yielded 3·61 per cent. of ash containing calcium, potassium, and phosphoric acid, but not iron or manganese. It was found to contain an indirect oxidizing enzyme. It yielded on hydrolysis 35·56 per cent. of pentoses and 42·67 of *d*-galactose, and thus closely resemble the gum of *Cochlospermum gossypium* in composition. C. A. M.

INORGANIC ANALYSIS.

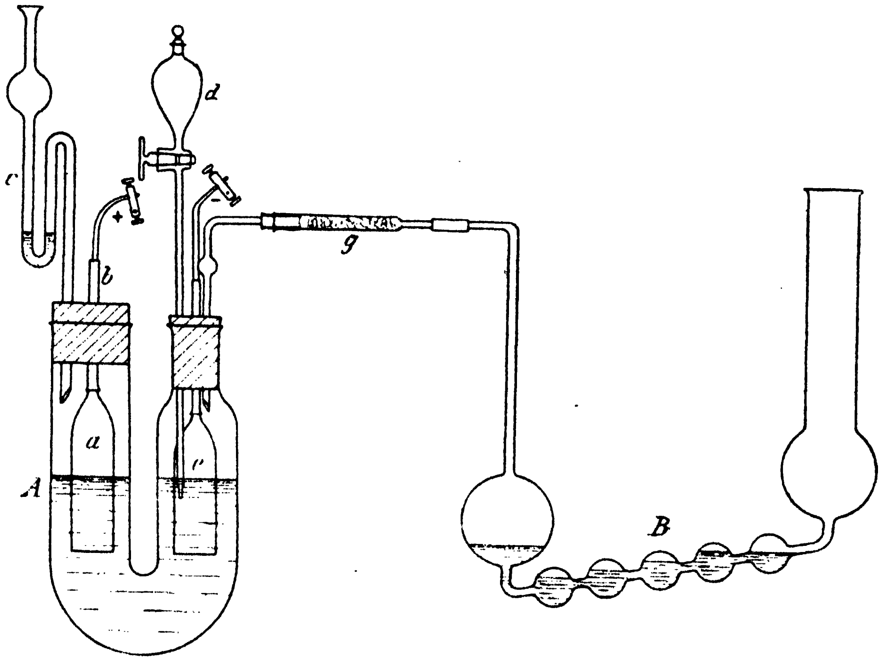
The Separation of Silver from Lead. H. Lidholm. (*Berichte*, 1905, xxxviii., 566-568.)—Silver is reduced from its solutions by various organic compounds, and notably by phenols, whereas lead forms sparingly soluble phenates with many of these. Hydroquinone gives no precipitate with lead, but precipitates silver quantitatively, provided the solution does not contain free mineral acid, a condition that can be insured by the addition of sodium acetate. The substance is dissolved, with the aid of tartaric acid if necessary, and the solution neutralized, treated with ammonium acetate, and brought to the boiling-point, any precipitated basic lead acetate being brought into solution again by the addition of acetic acid. It is then treated with a solution of hydroquinone in the proportion of 2 c.c. of a 4 per cent. solution to each 0·1 gram of silver, and the resulting precipitate of metallic silver collected, washed with water containing 5 per cent. of ammonium nitrate, dried, ignited, and weighed. If copper or bismuth are present, they too will be partially precipitated, and the ignited precipitate must then be dissolved in nitric acid, and the silver precipitated as chloride, the precipitate being freed from any bismuth by means of nitric acid, as directed by Fresenius. The presence of cadmium does not interfere with the results.

C. A. M.

The Prevention of Poisoning by Mercury Vapour. N. Tarugi. (*Gazzetta*, January 14, 1905; through *Pharm. Journ.*, 1905, lxxiv., 367.)—Finely-divided aluminium completely absorbs mercury vapour at the ordinary temperature, even when the vapour is largely diluted with air. On this fact the author bases a delicate test for the detection of mercury, and the action of the aluminium has been utilized (in the form of a respirator) as a preventive measure against poisoning by mercury vapour.

A. R. T.

The Electrolytic Determination of Small Quantities of Arsenic. C. Mai and H. Hurt. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 193-199.)—The authors employ the apparatus described below, in which the electrodes consist of pure sheet lead (see ANALYST, 1904, 268), and the liberated arseniuretted hydrogen is absorbed in silver nitrate solution. The decomposition vessel, *A*, is connected with the absorption bulbs, *B*, by means of the short tube, *g*, which contains pumice stone soaked in alkaline lead solution. The pointed upper ends of the cathode, *e*, and the anode, *a*, are cemented into glass tubes, *b*, passing through the stoppers of the apparatus. The funnel, *d*, has a capacity of 25 c.c., and the bore of its capillary stem is of such diameter that the solution under examination enters the apparatus drop by drop. The safety-tube, *c*, is filled with water. A 12 per cent. solution of pure sulphuric acid is placed in *A* up to the height indicated in the illustration, and 10 c.c. of $\frac{N}{100}$ silver nitrate solution are introduced into the absorption bulbs, *B*. The current may be obtained from the main, and regulated by a suitable resistance. After working the apparatus for some time, to prove that the acid and electrodes are free from arsenic, the solution to be tested is allowed to enter, without interrupting the current. The quantity of solution used should not exceed 10 c.c. Should arsenic be present, the silver solution is darkened within a few minutes, and after two, or at the most three, hours the reduction is completed. The contents of the bulbs are then filtered, the filter is washed with a little water, and the excess of silver in the filtrate titrated with $\frac{N}{100}$ thiocyanate solution as usual. One c.c. of $\frac{N}{100}$ silver nitrate solution is equivalent to 0·125 mgm. of arsenic or 0·1655 mgm. of arsenic trioxide. When the quantity of arsenic present is less than 0·02 mgm., the titration method fails, and the reduced silver must be determined colorimetrically. For minute traces of arsenic, down to 0·0005 mgm., the absorption bulbs may be replaced by the combustion tube of a Marsh apparatus and an arsenical mirror obtained in the usual manner.



W. P. S.

Electrolytic Analysis of Cobalt and Nickel. F. M. Perkin and W. C. Prebble. (*Chem. News*, 1904, xc., 307.)—Cobalt and nickel were deposited electrolytically from various solutions. With *cobalt*, solutions containing sodium hypophosphite gave very high results, the deposit containing about 5 per cent. of phosphorus. Ammonium oxalate and tartrate also gave somewhat high results, due to the presence of carbon in the deposit. Similar results were obtained with ammonium borate. The best results were given by solutions containing an alkali phosphate and a small quantity of free phosphoric acid. The procedure used was as follows: About 1 gram of $CoSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ (containing 14·94 per cent. of cobalt) was dissolved in 50 c.c. of water; 5 c.c. of a 5 per cent. solution of phosphoric acid and 25 c.c. of a 10 per cent. solution of ammonium, or, better, sodium dihydrogen phosphate, were then added, and the solution was made up to 130 c.c. and electrolysed, using a gauze-flag cathode. After about thirty minutes, 0·5 gram of hydroxylamine sulphate or chloride is added to dissolve a brown deposit which forms on the anode, and after the bulk of the cobalt has been deposited, it is advisable to add a few drops of ammonia to neutralize the acid liberated. With a current density of 0·3 to 1·8 ampère per 100 square centimetres the operation is complete in from three to three and a half hours at about 55° C.; it is best to start with a low current density, and to increase this after about one hour. The deposit obtained is brilliant; the error varies from about 0·14 to 0·45 per cent. on the cobalt.

With *nickel*, phosphate solutions gave unsatisfactory results. Solutions containing ammonia and ammonium borate, on the other hand, gave very good results. With ammonium tartrate solutions good results can be obtained if care is exercised, but they are apt to be erratic.

A. G. L.

The Analysis of Iron Sesquioxides. H. Cormimbœuf. (*Ann. de Chim. anal.*, 1905, 95, 96.)—About 0·5 gram of the sample is treated with an excess of hydrochloric acid to which are added a few crystals of potassium iodide to act as a reducing agent. After applying heat for a few moments, complete solution of all the iron is obtained, even in the case of the most refractory colcothars. The liquid is diluted with water, oxidized with nitric acid or bromine-water, and filtered from the residue of sand, etc. The iron in the filtrate is precipitated by means of ammonia, and the calcium and sulphuric acid subsequently determined in the usual manner.

C. A. M.

Determination of Phosphorus in Iron Ores. J. S. Rowland and Llewellyn J. Davies. (*Journ. Soc. Chem. Ind.*, 1904, xxiii., 1186.)—By incineration of the ore with magnesia, and subsequent treatment with dilute nitric acid, a solution is obtained which contains all the phosphorus, and the yellow molybdate precipitate can be subsequently titrated with a standard caustic soda solution, 1 c.c. of which is equivalent to 0·0001 gram of phosphorus. A quantity of pure yellow molybdate precipitate, dried at 100° C., and preserved in a stoppered bottle, is required.

One or two grams of the dry, finely-ground sample are well mixed with 1 gram of calcined magnesia free from phosphorus, and the mixture heated in a shallow porcelain dish to bright redness in a muffle-furnace. After cooling, the mixture is dissolved in 25 c.c. of nitric acid (equal parts acid of 1·42 specific gravity and water) by boiling the liquid for ten minutes. After standing, the clear liquid is filtered into a flask. Twenty-five c.c. of water are added to the residue, boiled for five minutes, and filtered through the same filter, washing the residue until free from acid. Five c.c. of ammonia are next added and the solution boiled, and then 50 c.c. of ammonium molybdate solution (E. F. Woods' 1888 formula), and the mixture shaken for ten minutes. The liquid is immediately filtered, the precipitate washed five times, each with 1 per cent. nitric acid and potassium nitrate solution (1 gram per litre), and then until free from acid. The filter-paper and precipitate are transferred again to the flask, a known quantity of standard caustic soda added (10 to 20 c.c.), and the liquid shaken, when the precipitate will readily dissolve. After diluting the liquid with water to about 50 c.c., the excess of caustic soda is titrated back with standard nitric acid solution, using phenolphthalein as indicator. From the number of c.c.'s of caustic soda required the amount of phosphorus present can be deduced.

For the preparation of the standard solution of alkali, 7·2 grams of caustic soda are dissolved in 100 c.c. of water, a saturated solution of baryta-water added until no further precipitation takes place, the liquid filtered, and the solution made up to 2 litres. The standard nitric acid solution is made by diluting 10 c.c. of 1·42 nitric acid to 2 litres with water. The acid and alkali are then titrated against each other, and the stronger one diluted until they are of the same relative strength. The phosphorus equivalent of the caustic soda is next determined by titrating it against 0·100 gram of the pure yellow molybdate precipitate (= 0·00163 gram phosphorus). If the caustic soda solution is too strong, it should be diluted with water until 1 c.c. = 0·0001 gram phosphorus. A similar quantity of water should then be added to the standard acid solution to make it exactly equivalent to the caustic soda solution. The presence of silica in the yellow precipitate does not affect the results, which are stated to be very accurate, while the process only requires forty minutes. A. R. T.

Estimation of Sulphur in Iron Ores, Slags, Limestone, etc. H. Hartwigsson. (*Chem. Zeit. Rep.*, 1905, xxix., 4.)—The method employed is founded on the fact that some sulphur-containing iron ores, when heated in a current of hydrogen, give off their sulphur as sulphuretted hydrogen, and though others do not, in all cases any sulphur not so converted is left in such a condition that sulphuretted hydrogen is evolved on treatment with hydrochloric acid.

The analysis is conducted as follows: 1 to 5 grams of the finely powdered sample are introduced in a porcelain boat into a combustion tube. After air has been expelled from the apparatus the tube is raised to a red heat and maintained so for three-quarters to one hour. A stream of hydrogen, purified in the usual way, is then passed through the tube, and the issuing gas passed through two Erlenmeyer flasks of 200 c.c. capacity, containing 30 to 40 c.c. of a cadmium acetate solution, prepared by dissolving 25 grams cadmium acetate in a mixture of 200 c.c. concentrated acetic acid and 800 c.c. water. A yellow precipitate of cadmium sulphide is usually obtained. The tube is then cooled in a current of hydrogen, the residue transferred from the boat to a filter paper; the filter paper is carefully rolled up, and placed in a flask, provided with a reflux condenser, to which are connected the two Erlenmeyers used before. The air is expelled by carbon dioxide, and 150 c.c. hydrochloric acid (1 : 2) let in through a dropping funnel; the flask is heated, and, after the evolution of sulphuretted hydrogen has ceased, the contents of the two Erlenmeyers are mixed.

An excess of an iodine solution (3·97 grams iodine and 10 to 15 grams potassium iodide in 1,000 c.c., 1 c.c. = 0·0005 gram sulphur) is added, the cadmium sulphide then decomposed by the addition of 10 to 20 c.c. hydrochloric acid, and the excess of iodine over that required to act with the liberated sulphuretted hydrogen found by titration with thiosulphate of soda (7·77 grams in 1,000 c.c., 1 c.c. = 1 c.c. of iodine solution used).

The whole process requires about two hours, and the trial analysis made by the author gave results agreeing well with those obtained by the direct oxidation method.

E. K. H.

Determination of Sulphur in Pyrites by Lunge's Method. H. Salvin Pattinson. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 7.)—Lunge's method is easier and less expensive to work than that of Silberberger, but the latter gives accurate results when carefully carried out. The author has made experiments with a view to overcoming the following sources of error which may arise in Lunge's process: (1) Retention of iron by the barium sulphate precipitate; (2) solubility of barium sulphate in the filtrate and washing water; (3) co-precipitation of barium chloride with the barium sulphate. Lunge avoids (1) by first precipitating the iron with ammonia and filtering off the precipitated ferric hydroxide, but this procedure introduces the possibility of carrying down sulphur in the form of basic ferric sulphate.

The author finds that the quantity of hydrochloric acid present in the sulphate solution not only influences the physical character of the barium precipitate, but also affects the proportion of barium chloride carried down with it. Further, the amount of ammonia to be added in excess, to precipitate the iron, decides the formation of the basic ferric sulphate. The following mode of operating is recommended: After the oxidation of the pyrites as directed by Lunge, and subsequent separation of the silica, etc., the liquid is heated to 70° C., and the ferric hydroxide precipitated by ammonia, an excess of 5 c.c. of 0·880 ammonia being employed. The liquid is kept at 70° C. for ten minutes and then filtered, and the precipitate washed in the usual manner. The washing requires from a half to one hour, and the filtrate and washings then measure about 350 to 400 c.c. Without concentration the liquid is made neutral to methyl-orange by hydrochloric acid, an excess of 1 c.c. of hydrochloric acid of 1·17 specific gravity added, and the sulphate precipitated from the boiling solution by the addition of 20 c.c. of boiling barium chloride solution (10 per cent.). A blank experiment should be made with the materials employed, and the amount of barium sulphate found deducted, in which case the necessity of determining the proportion of sulphate lost in the wash-waters is avoided, since the same error operates both in the blank experiment and the actual determination. Lunge agrees with the author's detailed method of working.

A. R. T.

The Estimation of Sulphur or Sulphuric Acid in Pyrites by Means of Benzidine Hydrochloride. G. v. Knorre. (*Chem. Ind.*, 1905, xxviii., 2; through *Chem. Zeit. Rep.*, 1905, xxix., 29.)—The author finds that the method suggested by Müller (*Berichte*, 1902, xxxv., 1587) and further elaborated by Raschig (*Zeit. Angew. Chem.*, 1903, xvi., 617), in which the sulphuric acid in the benzidine compound formed is directly titrated, gives excellent results in the analysis of pyrites. The presence of ferric compounds, especially ferric chloride, was observed to oxidize the benzidine compound, discolouring the solution and rendering titration difficult. The iron should therefore first be reduced or separated. Hydrazine hydrochloride and sulphurous acid are satisfactory reducing agents; whilst tartaric and citric acids and their salts give unreliable results.

H. A. T.

Determination of Zinc in Blendes, etc. H. Salvin Pattinson and George C. Redpath. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 228.)—The authors have examined the three following methods: (1) Von Schulz and Low's Method (*Journ.*

Soc. Chem. Ind., 1892, p. 846); (2) Von Berg's Method (Dittmar's "Quantitative Analysis"); and (3) Lewis's Method (ANALYST, xxviii., 93), and consider that the three methods give closely-agreeing results, the most rapid and convenient being the first. Of the two others, Von Berg's is preferable, since the sulphide precipitate is denser and can be more readily washed; and if cobalt is present, it is completely separated in Method 2, while the presence of this element renders Method 3 inaccurate. Method 1 may be employed in the separation of zinc from iron ores, and is more rapid than the acetate method. A. R. T.

Use of Tannic Acid in the Determination of Alumina. Robert E. Divine. (*Journ. Soc. Chem. Ind.*, 1905, xxiv, 11.)—Owing to the difficulty of filtering and washing alumina precipitates free from chlorides, the author has employed tannic acid and ammonia to separate alumina from its solutions. To a solution containing about 0·1 gram of alumina, 2 c.c. of a 2½ per cent. solution of tannic acid, some ammonium chloride, and ammonia in slight excess, are added, and the liquid boiled until nearly all the ammonia is expelled. By this method the alumina is obtained in a form which allows of its rapid filtration, especially if suction be used; whilst it may be freed from chlorides by washing somewhat, redissolving in hydrochloric acid, reprecipitating with ammonia, and well washing the precipitate. The slight trace of alumina sometimes obtained on evaporating the filtrate and rendering alkaline with ammonia may be added to the main quantity, although its amount is practically negligible.

Even in the presence of considerable quantities of lime, magnesia, and chlorides of the alkali metals, the separation of alumina by this method is very complete and satisfactory, whilst the presence of tannic acid does not interfere with the subsequent determination of the lime and magnesia in the filtrate. Large quantities of ferric hydroxide with the alumina do not prevent the ready filtration of the precipitate, although pure ferric hydroxide does not filter well in presence of tannic acid.

A. R. T.

A New Method of Determining Molybdenum Trioxide and Vanadium Pentoxide in the Presence of each other. B. Glasmann. (*Berichte*, 1905, xxxviii., 600-603.)—A solution of molybdium trioxide in hydrochloric acid is reduced by either zinc or magnesium to molybdenum sesquioxide, whereas vanadium pentoxide under the same conditions is reduced by zinc to the dioxide, but by magnesium to the trioxide. Hence, if a solution containing the mixed oxides be divided into two parts, one of which is boiled in a flask fitted with a Bunsen valve, with zinc and hydrochloric acid, and the other with magnesium and hydrochloric acid, a subsequent titration of the two solutions with permanganate solution will give the amounts of vanadium and molybdenum.

The difference between the amounts of permanganate consumed in the two titrations thus gives the amount required to convert the vanadium from the dioxide into the trioxide ($2KMnO_4 = 5V_2O_2$). In the analysis from 0·25 to 0·5 gram of the substance is boiled for one to one and a half hours with 40 to 45 c.c. of hydrochloric acid (specific gravity 1·19) and zinc and magnesium respectively. The solutions are subsequently poured into large basins containing 300 c.c. of water with

10 grams of manganese sulphate in solution, and titrated with $\frac{N}{10}$ potassium permanganate solution with continual agitation. C. A. M.

The Reduction of Molybdenum Compounds in Sulphuric Acid Solution by Means of Magnesium. B. Glasmann. (*Berichte*, 1905, xxxviii., 604, 605.)—Molybdenum trioxide can be reduced by means of magnesium, and the molybdenum then determined by titration with standard permanganate solution. For this purpose the substance is heated for thirty to forty-five minutes with magnesium and dilute sulphuric acid (1 : 5) in a flask fitted with a Bunsen valve. The contents are then transferred to a large basin, diluted with about 300 c.c. of water acidulated with sulphuric acid, and titrated with $\frac{N}{20}$ permanganate solution at a temperature of 30° to 40° C. C. A. M.

The Quantitative Estimation of Tellurium. A. Gutbier. (*Sitz. Ber. of the Soc. Phys. Med., Erlangen;* through *Chem. Zeit. Rep.*, 1904, xxviii., 353.)—The author discusses the conditions necessary for successful reduction and precipitation of tellurium by means of hydrazine, and considers that the solution should be as concentrated as possible. It should contain no nitric acid, and recently precipitated tellurium must also be avoided.

Hydrazine hydrate was found to be the best general precipitant. H. A. T.

Note on the Quantitative Estimation of Tellurium. A. Gutbier and W. Wagenknecht. (*Journ. Pract. Chem.*, 1905, lxxi., 54; through *Chem. Zeit. Rep.*, 1905, xxix., 28.)—Frerichs (*Journ. Pract. Chem.*, 1905, lxvi,. 54) suggests a process for the estimation of tellurium by means of potassium iodide and sulphurous acid in the presence of sulphuric or hydrochloric acid. From a number of experiments the authors conclude that this method is quite unreliable, owing to the fact that some tellurium tetra-iodide is always formed. H. A. T.

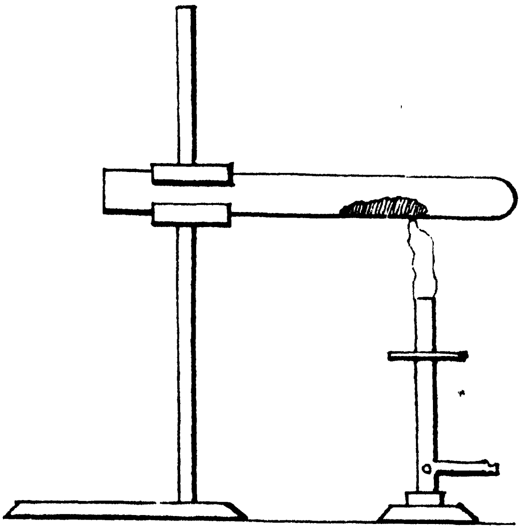
Practical Hints for the Performance of an Easy and Convenient Estimation of Potassium. Friedrich Klinkerfues. (*Chem. Zeit.*, 1905, xxix., 77.)—The author disclaims any fresh principle in his method, which is simply a modification of the process based on the reduction of potassium platinichloride to platinum by formic acid. The modification consists in reducing to platinum in a weighed platinum dish and washing by decantation without transferring the precipitated platinum to a filter, as the author finds it adheres to the dish sufficiently to prevent any loss.

The modified process is carried out as follows: From 0·5 to 1·0 gram of substance, according to its potassium content, is dissolved in water, excess of platinic chloride solution added, and the whole evaporated to dryness on the water-bath. A little water is added to the residue, which is then rubbed up with a stirring rod, and extracted with alcohol until all matter soluble in that menstruum is removed, the alcoholic washings being passed through a filter in order to retain any particles of potassium salt which may be carried over. The residue in the dish is then dissolved in boiling hot water, and passed through the before-mentioned filter into a tared platinum dish, a couple of drops of formic acid added, the whole evaporated to dryness, and the residue repeatedly extracted with hot water until no more chloride or sulphate occurs in the washings—in some cases it may be necessary to first digest with 5 per cent. HCl for half an hour. The platinum basin is then gently ignited, allowed to cool in a vacuum desiccator and weighed, and the platinum calculated to K_2O.

The author finds this method accurate with an error of less than 0·3 per cent. He recommends it especially for cases where potassium is to be estimated in superphosphate, etc.

Experiments are in hand to determine whether any cheaper material can be substituted for the platinum basin. E. K. H.

Eschka's Method of Estimating Sulphur. C. Bender. (*Zeits. f. angew. Chem.*, 1905, xviii., 293.)—The author has made experiments with a view to comparing the merits of Sauer's method for determining sulphur in coal with that of Eschka, and has obtained identical results, even when Eschka's method was conducted over a gas-flame.



The author modified Sauer's process by using platinum foil in place of a combustion-tube for the combustion.

Eschka's method, which was also modified so as to prevent the combustion gases from coming into contact with the heated mixture of coal and soda-magnesia, is conducted as follows:

The mixture is placed towards the closed end of a tube of difficultly fusible glass, about 18 centimetres long and 2·3 to 3 centimetres in diameter, which is left open at the other end. Having been completely dried in an air-bath, it is clamped horizontally in a stand (see figure), and is heated by a Bunsen burner capable of giving a powerful flame. Heat is first applied to the border of the mixture, and the flame gradually moved along the tube, which is rotated from time to time, and the heating continued until the mass is white. On the completion of the combustion the holder is lowered, the tube somewhat inclined, and a porcelain dish placed directly under it. On cooling, the tube usually breaks up and falls into the dish. The residue is treated as in the original process of Eschka.

The author recommends that the tubes should be obtained from a professional glass-blower. E. K. H.

A New Iodometric Method of Determining Alkali Heptamolybdates. B. Glasmann. (*Berichte*, 1905, xxxviii., 193, 194.)—Iodine is precipitated from an aqueous solution of potassium iodide and iodate, through the conversion of the heptamolybdate into a neutral salt and free molybdic acid, which then reacts with the mixture. Thus—

$$(a)\ 3(NH_4)_6Mo_7O_{24} + 12H_2O = 9(NH_4)_2MoO_4 + 12H_2MoO_4;$$
$$(b)\ 20KI + 4KIO_3 + 12H_2MoO_4 = 12K_2MoO_4 + 12I_2 + 12H_2O.$$

In the analysis from 0·2 to 0·3 gram is mixed with 0·5 gram of potassium iodide, 0·1 gram of potassium iodate, and distilled water in the distillation flask of Bunsen's iodine apparatus. The mixture is then distilled, and the iodine collected in the receiver and titrated with standard thiosulphate solution. One part by weight of ammonium heptamolybdate, $(NH_4)_6Mo_7O_{24} + 4H_2O$, is equivalent to 0·822 part of iodine. The analytical results show that this method is very accurate. C. A. M.

Some Sources of Error in Sulphur Determinations. John Pattinson and J. T. Dunn. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 10.)—Some of the red indiarubber stoppers used for wash-bottles are acted upon to a serious extent by boiling water or steam, yielding sulphuric acid to the water. This sulphuric acid has its source in the oxidation of the sulphur in the rubber or in the metallic sulphides, such as antimony sulphide, added to the rubber. Such stoppers may introduce a serious error in the determination of sulphur in iron and steel. Soluble unoxidized sulphur compounds are sometimes present in commercial barium chloride, due no doubt to the fact of its being prepared from heavy spar by reduction to sulphide and treatment with hydrochloric acid. Many samples of barium chloride, when dissolved in water and oxidized with nitric acid or bromine and boiled, yield a precipitate of barium sulphate. A. R. T.

The Diphenylamine Test for Nitric Acid. G. Frerichs. (*Arch. Pharm.*, 1905, ccxliii., 80; through *Chem. Zeit. Rep.*, 1905, xxix., 84.)—Ferric salts and chromates react with diphenylamine in a similar manner to nitric acid, and these compounds, if present, must be eliminated from the substance to be tested before applying the reaction. This is best carried out by extraction with ether.

The substance is shaken in a test-tube with about 10 c.c. of dilute sulphuric acid and 20 c.c. of ether; any precipitate is allowed to settle, and 2 to 3 c.c. of the ethereal solution filtered through a dry paper. Five to ten c.c. of concentrated sulphuric acid are then carefully added, when any nitric acid present will give the characteristic reaction.

Should the ethereal solution contain iodine, bromine, or chromic acid, it will be yellow in colour. Iodine and bromine do not affect the test. Chromic acid is eliminated by shaking with a little aqueous sulphurous acid, filtering the ether solution, and testing as before. H. A. T.

The Differentiation of Nitric and Nitrous Acids by Means of Diphenylamine. P. Raikow. (*Oest. Chem. Zeit.*, 1904, vii., 557.)—An exhaustive paper, in which the various methods for the identification of nitrates in the presence of nitrites are discussed. The author considers that diphenylamine is the only satisfactory reagent, and suggests an improved method of applying this reaction.

0·5 c.c. of a 2 per cent. solution of diphenylamine in sulphuric acid (specific gravity, 1.78) is introduced into a flat-bottomed porcelain dish, and a single drop of the solution to be examined is allowed just to come into contact with the reagent. Nitrate solutions, in strength from twice normal to $\frac{N}{100}$, give a blue colour, becoming brown or black on standing. Weaker solutions, down to $\frac{N}{10000}$, give no immediate colour, but a blue appears in from four to ninety seconds, according to the extent of dilution of the solution, and, after increasing for a few minutes, slowly disappears. Nitrite solutions, in strength from normal to $\frac{N}{10}$, react similarly to nitrates weaker solutions; give an immediate violet colour, rapidly becoming blue and disappearing. This violet colour is distinct with solutions as weak as $\frac{N}{50000}$ ($NaNO_2$).

These reactions are suggested as affording a certain indication as to whether nitric or nitrous acid, or both, are present. With a suitably dilute solution containing nitrate only, no immediate reaction is obtained, but a blue colour, gradually increasing in intensity, shows after a few seconds. The colour disappears slowly.

A nitrite, on the other hand, gives a violet colour immediately, which rapidly becomes blue, and disappears in from two to ten minutes.

A sufficiently dilute mixture of both acids gives an immediate violet colour (nitrite), becoming pale blue, which quickly disappears. A blue colour is then gradually formed, which becomes more intense, and only slowly vanishes (nitrate). The exact degree of dilution necessary to give a satisfactory reaction must be found by experiment.

Dilution of a solution containing only nitrate or nitrite mainly affects the intensity, etc., of the reaction, by progressive dilution of a mixed solution, a point is finally reached at which the two reactions are quite distinct.

Using phosphoric acid as solvent for the diphenylamine, the reaction for nitrites is unaltered. Nitrates acquire a quite characteristic reaction. At the point of contact, numerous black points are observed, the solution itself remaining colourless. These points gradually expand and merge, a blue solution being formed. This reaction however, is only noticed in strong solutions (normal to $\frac{3}{4}$ normal KNO_3).

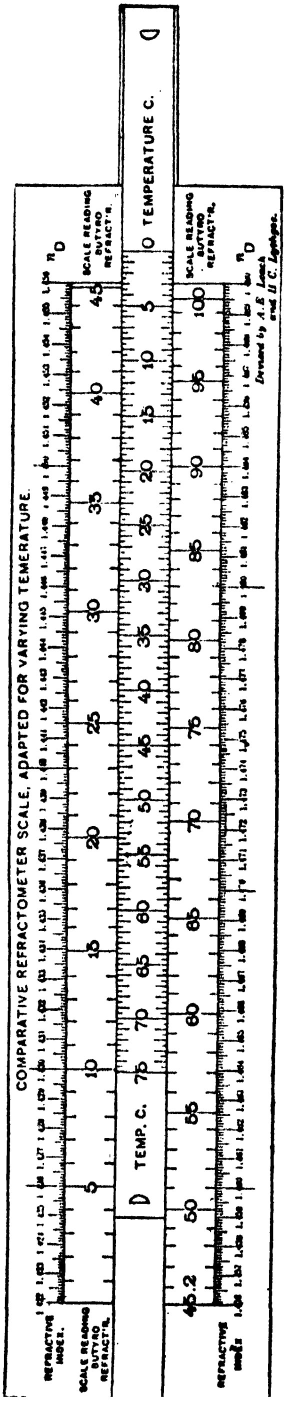
The smallest quantity of KNO_3 which can be detected by the phosphoric acid solution is 0·0025 gram. The reaction with $NaNO_2$ is definite when as little as 0·00006 mgm. is present. Any colour, therefore, after sufficient dilution is a sure indication of nitrous acid.

A solution of diphenylamine in acetic or formic acid proved quite unsuitable for the purpose.
H. A. T.

Preparation of Volumetric Solutions. E. C. Worden and John Motion.

(*Journ. Soc. Chem. Ind.*, 1905, xxiv., 178.)—The paper consists of a number of valuable tables showing the relation of specific gravity to composition in the cases of sulphuric, hydrochloric, and oxalic acids, for use in the preparation of volumetric solutions.
A. R. T.

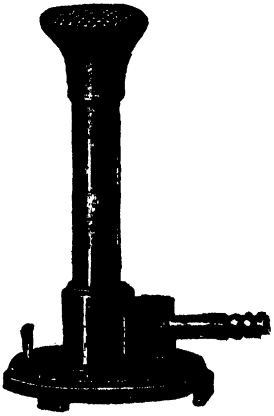
APPARATUS.


COMPARATIVE REFRACTOMETER SCALE, ADAPTED FOR VARYING TEMERATURE.
0 TEMPERATURE C.
TEMP. C.
REFRACTIVE INDEX.
SCALE READING BUTYRO REFRACT'R.
Devised by A. E. Leach and H. C. Lythgoe.

A Comparative Refractometer Scale for use with Fats and Oils. A. E. Leach and H. C. Lythgoe. (*Jour. Amer. Chem. Soc.*, 1904, xxvi., 1193-1195.)—The Zeiss butyro-refractometer has an arbitrary scale from 0 to 100, covering the indices of refraction from 1·4220 to 1·4895, which scale is not uniform with the values of n_D. Thus the difference from 0 to 10 in indices of refraction is 0·0080, and from 90 to 100 is 0·0055, whilst the variation for 1° C. on the refractometer is 0·46 for readings between 0 and 10, and 0·66 for readings from 90 to 100. The best method of obtaining scale readings at different temperatures is to convert them into indices of refraction, apply the correction (0·000365 for each degree), so obtaining the indices at the required temperature, and then to recalculate the figures into scale readings. To avoid this long calculation, the authors have devised the slide rule illustrated. By the use of this instrument readings may be obtained on the butyro-refractometer scale at different temperatures, without first transforming them into indices of refraction; readings of the latter may be obtained at different temperatures without calculation, and values on either scale can be readily transformed into values on the other. The authors are making arrangements for the manufacture of the instrument, but pending this prints of the scale may be obtained by applying to the authors at Room 501, State House, Boston, U.S.A. These can be cut and used like a slide rule.

W. P. S.

A Simple Bunsen Burner. F. Allihn. (*Chem. Zeit.*, 1905, xxix., 34, 35.)—This burner is the same in principle as that previously described by the author (ANALYST, 1900, 138). The vertical tube of the burner is open at the bottom, and the jet is done away with. A gauze cover is provided at the top, so that a uniformly hot flame is obtained. This flame is particularly suitable for heating platinum vessels, as the objectionable "inner cone" of the ordinary Bunsen flame is absent. The small upright arm shown in the illustration at the left of the base is the handle of the air-



regulator, but the burner is also made without this device. The burner may be obtained from Warmbrunn, Quilitz, and Co., Berlin, N.W. W. P. S.

A New Form of Eggertz Tube for the Determination of Carbon in Steel and Iron. H. Schumacher. (*Chem. Zeit.*, 1905, xxix., 35.)—The tube shown in Fig. 1 really consists of two tubes separated by a milk-glass partition. Each is graduated, and has a capacity of 20 c.c. The tube *b* holds the standard steel solution, and is closed by a cork; whilst the tube *a* contains the steel solution under examination. When it is necessary to dilute this, the tube is closed by means of the finger and shaken. The milk-glass partition prevents the coloration in one tube influencing that in the other.

Another form of tube is shown in Fig. 2, and is suitable for other colorimetric determinations. The tubes are sold in sets, and have one side flattened. In each set one tube has its flat side made of milk-glass, and is intended to hold the standard solution. By reason of their flat sides the tubes may be brought close together, and the comparison of the colour of their contents is rendered more easy. The tubes are sold by A. Eberhard, Berlin. W. P. S.

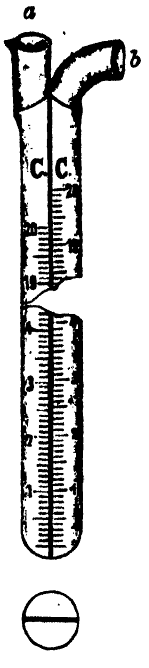


FIG. 1.

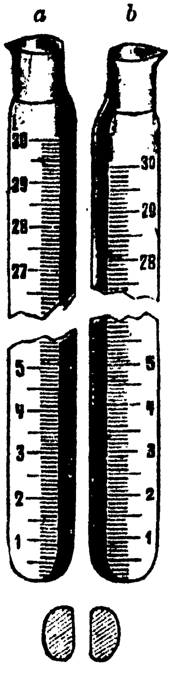
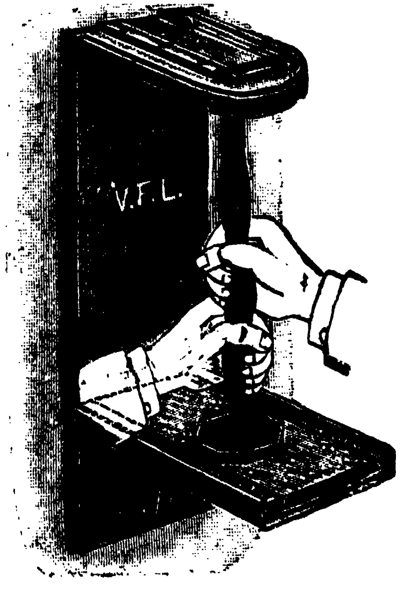


FIG. 2.

A Practical Mechanical Mortar. (*Chem. Zeit.*, 1905, xxix., 56.)—The instrument as illustrated is supplied by the "Vereinigten Fabriken für Laboratoriumsbedarf," Berlin, N. It consists of an agate mortar fixed into a wooden stand, which also has an upper portion, in which is a socket for the wooden handle, in which is set the agate pestle. The arrangement thus allows both hands to be used for the grinding. A slide on the upper guiding place allows more or less lateral play as required. E. K. H.



REVIEWS.

CHEMICAL TECHNOLOGY AND ANALYSIS OF OILS, FATS, AND WAXES. By DR. J. LEWKOWITSCH, M.A., F.I.C. Third Edition. (London: Macmillan and Co. Two vols. Price 36s. net.)

For many years the subject of oils and fats was one of the most neglected branches of analytical chemistry, for the problems it presented were looked upon as almost incapable of solution. Now the reverse is the case, and every month sees the publication of papers dealing with different processes or with the characteristics of individual fats and oils. And so important are the industrial applications of these products that it is essential for the analytical chemist to keep in touch with the new facts recorded about each of them. Hence it is not surprising that the present work, which in its first edition was contained in one volume of moderate size, has now expanded into two volumes containing 1,152 pages, and has only been kept within even these limits by the rigorous exclusion of matter which the author regarded as possessing only historical interest. This was, doubtless, an inevitable necessity, but at the same time it detracts from the completeness of the work, even from the practical point of view, since many methods that we have abandoned as apparently useless may yet form the starting-point for new researches introducing other principles.

The value of the book has been considerably enhanced by the introduction of chapters dealing with the technology of the subject; but here again the virtue of compression has been carried too far, and insufficient space has been given to the description of apparatus and manufacturing processes.

The arrangement of the book differs somewhat from that of the earlier editions. The first three chapters deal with the general properties and composition of oils and fats and the theory of saponification, and are followed by chapters describing the general physical and chemical methods of examination. The second volume opens with a chapter on the commercial preparation of oils and fats, which is followed by a systematic description of the individual products and two chapters dealing with the technology of every description of fat, and the work concludes with a good index.

The book has been brought thoroughly up to date, and is a most valuable textbook for the analytical laboratory. Perhaps Chap. XIV. will still be found the most useful to the analyst, for the excellent tables of values, which formed a striking feature of the first edition, have been greatly enlarged and extended so as to include all known fats.

The book is well printed and is singularly free from errors, although there is one slip that is constantly repeated. The author, in writing about the insoluble bromides given by certain oils, describes them as "hexabromides," whereas it has been shown that they do not contain the percentage of bromine required by the hexabromide of linolenin, and all the evidence points to their being bromides of mixed glycerides containing as one of their constituents the glyceride of linolenic or jecoric acid.

C. A. M.

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HIGH COURT OF JUSTICE.

KING'S BENCH DIVISION.

(*From the " Times " of Thursday, April* 18, 1905.)

(Before the Lord Chief Justice of England, Mr. Justice Kennedy, and Mr. Justice Ridley.)

Smith *v.* Savage.

This was a case stated by the justices of Wiltshire sitting at Marlborough, and raised a question under the Food and Drugs Act.

The respondent, Henry Edward Savage, a grocer, was charged at the instance of the appellant, an inspector under the Food and Drugs Act, with having, on August 9, 1904, unlawfully sold to the appellant, to his prejudice as the purchaser thereof, four penny packets of cream of tartar, which were not of the nature, substance, and quality of the article demanded, such cream of tartar containing lead in the proportion of ¾ grain per pound, contrary to Section 6 of the Food and Drugs Act. On August 9, 1904, the appellant visited the respondent's shop, and saw there some packets of cream of tartar. He asked the respondent if he sold cream of tartar, whereupon the respondent produced a box containing packets labelled "Finest cream of tartar, 98 per cent. bitartarate of potassium, B.P., 1898." The appellant asked for four packets, which were all similar in size and outward appearance and label, and were taken from the same box. He paid fourpence for them, and told the respondent that the purchase had been made for the purpose of analysis. The appellant emptied the contents of the four packets into one place, and then divided the whole of the contents or matter into three parts, and sealed them up. One of these three packets was sent to the county analyst, another handed to the respondent, and the third packet was retained and produced to the justices by the appellant. The appellant said that cream of tartar mixed with bicarbonate of soda was used in baking-powder, and also in the preparation of cakes, etc. The result of the analysis as above stated was proved, whereupon the appellant asked for a conviction. The respondent's solicitor contended that the appellant made four separate purchases, each packet being an article, and that the appellant did not by mixing together the contents of the four packets, and then dividing the substance so mixed into three parts, comply with Section 14 of the Food and Drugs Act, 1875, and, in support of this argument, the case of Mason *v.* Cowdary (16 *The Times* L.R., 484 ; 1900, 2 Q.B., 419) was cited. For the appellant it was contended that the facts were not similar to those in Mason *v.* Cowdary, and that Section 14 had been properly complied with, inasmuch as the entire purchase had been properly divided into three parts as the Act directed. It was also pointed out that in Mason *v.* Cowdary there was no mixing and division of the entire purchase. The justices dismissed the summons, and the question for the opinion of the Court was whether they were right in so doing.

Mr. Holman Gregory, for the appellant, submitted that the justices were wrong in dismissing the summons. All the packets were labelled in the same manner, and given to the appellant as cream of tartar, not as four packets. Making up the article in packets was only a means of measuring the quantity. No question could have arisen if the appellant had asked for fourpennyworth of cream of tartar. The case of Mason *v.* Cowdary was not in point. That was a case in which six bottles of camphorated oil were bought ; there was no division of the contents of any one bottle, but two bottles were sent to the analyst, two given back to the seller, and two retained by the inspector. That was held not to be a compliance with Section 14, but it was obvious that the oil might be of quite different quality in the different bottles. That was not the case as to the packets of cream of tartar, which were all labelled in the same way and taken from the same box.

Mr. Bonsey, for the respondent, contended that the justices were right. There had been four separate purchases, and not one purchase. Suppose a person were to purchase several loaves of bread, one of the loaves would have to be divided into three parts in order to comply with the Act. Mason *v.* Cowdary was directly in point; in that case Mr. Justice Darling said that "six bottles are six articles." In that case it might, with equal force, have been contended that oil, and not six bottles of oil, had been asked for by the inspector; so here it could not be said that merely cream of tartar was demanded; the request was for four packets of it. There was no reason why one of the packets should not have been divided into three parts. It had been suggested that one packet was too small to permit this being done; that was not so, and, moreover, whatever the quantity might be could make no difference, as the Act, which required division into three parts, must be complied with.

The Lord Chief Justice, in giving judgment, said that on the whole he thought the appeal must succeed. Some question, he thought, might arise in the case of different articles not of a similar name or articles coming from different people being mixed together in the shop; that, however, was not the case with which they had to deal. In the case before them the appellant asked for cream of tartar, which, for the purpose of measurement, was put up in penny packets. He said he would take four packets, and they had to say whether because four packets of the same article, labelled in the same way, and sold at the same time as cream of tartar, were mixed together and then divided into three parts was a good objection to the analysis that followed. He was of opinion that it was not a good objection. The case was different from Mason *v.* Cowdary; it must, therefore, go back to the justices to proceed in the matter.

Mr. Justice Kennedy and Mr. Justice Ridley concurred.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST OF THE APRIL EXAMINATIONS.

Of six candidates who entered for the Intermediate Examination, three passed: J. D. Kettle, B.Sc. (Lond.), Elison A. Macadam, and R. Simmons. In the Final Examination for the Associateship (A.I.C.), of seven examined in the branch of Mineral Chemistry, three passed: J. Alexander, B. O'Shaughnessy, Assoc.R.C.Sc. (Lond.), and E. Rhodes, B.Sc. (Vict.); of five in Organic Chemistry, three passed: S. J. M. Auld, Ph.D. (Würzburg), H. W. Goodwin, and R. Robison; and of eight who entered in the branch of the Analysis of Food and Drugs and of Water, including an examination in Therapeutics, Pharmacology, and Microscopy, the following six passed: J. H. Barnes, B.Sc. (Birm.), J. W. Brisbane, D. Gair, B.Sc. (Lond.), H. G. Harrison, B.A. (Cantab.), R. Park, and J. Race. One candidate passed an examination for the Fellowship (F.I.C.): A. E. Brown, B.Sc. (Lond.). The examiners in chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. The examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., F.I.C.

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The Late Professor Tichborne.

With regret we have to announce the death of an old and distinguished member of the Society, Professor C. B. C. Tichborne, which took place on May 1. A full notice of his scientific career will appear in our next issue.

Erratum.—Page 110, eighth line from bottom, for February 1 *read* "March 1."

JUNE, 1905. Vol. XXX., No. 351

THE ANALYST.

OBITUARIES.

PROFESSOR TICHBORNE.

PROFESSOR CHARLES ROBERT TICHBORNE, whose death we briefly announced last month, was born in 1839 in Birmingham, where he received his early education. At the age of fourteen he was apprenticed as chemist at a chemical manufactory, where he stayed six years, and at the end of this time became a student under Hofmann at the Royal College of Science. In 1859 he was appointed head of the laboratories of the Irish Apothecaries' Hall, which position he held up to the time of his death. Here he at once commenced a series of investigations, which led to many valuable suggestions being made as to the preparation of a number of pharmaceutical preparations. He also did good work in analytical chemistry, and, amongst other things, was the inventor of a process for collecting and liquefying carbonic acid gas in breweries. He became Lecturer on Chemistry at the Carmichael School of Medicine in 1872; was one of the original members of the Pharmaceutical Society of Ireland, which commenced its career in 1875, succeeding Sir D. Corrigan, Bart., as president in 1878. In 1887 Professor Tichborne qualified as a medical practitioner, and in 1896 was appointed representative of the Irish Apothecaries' Hall on the Medical Council. He was a Fellow of the Chemical Society, a Fellow of the Institute of Chemistry from its foundation, also an original member of the Society of Public Analysts, and held the appointment of Public Analyst for the county of Longford. Dr. Tichborne was a cultured, kindly, and good-hearted gentleman, whose memory will long survive in the minds of his colleagues, and especially in the hearts of those who were fortunate enough to be his students, for to them he was a most painstaking teacher.

MR. WILLIAM ACKROYD.

WITH regret we have to announce the death at Halifax, on May 9, of Mr. W. Ackroyd, at the age of fifty years, who had been Public Analyst for that borough for the last nineteen years, and was also teacher of Chemistry at the Halifax Technical School. Mr. Ackroyd was a Fellow of the Institute of Chemistry, also of the Chemical Society, and a member of the Society of Public Analysts. He was a keen student in chemistry and physics, and published a number of original papers in the *Chemical Society's Transactions*, the British Association Reports, and the *Chemical News*: "Researches on Moorland Waters on the Origin of Combined Chlorine," *J. C. S. Trans.*, 1901, p. 673; papers relating to the circulation and distribution of chlorides in natural waters ; to the distribution of the elements in relation to their atomic weights ; and to the colour effects and the action of radium rays on the haloid salts of the alkali metals, etc.

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THE VOLUMETRIC DETERMINATION OF REDUCING SUGARS.

By ARTHUR R. LING, F.I.C., AND THEODORE RENDLE.

(*Read at the Meeting, February* 1, 1905.)

THERE are but few subjects in analytical chemistry about which more has been written than the determination of sugars by the cupric reduction method. Every year a number of papers appear in the scientific journals describing certain fresh modifications in the carrying out of the process. The majority of these, however, add little to our knowledge ; whilst in not a few of the more recent contributions to the subject it would seem that the authors have either an insufficient acquaintance with the literature, or that they do not properly appreciate the degree of accuracy requisite in those branches of chemical industry in which sugar determinations play a prominent part. It is, therefore, not without some reluctance that we venture to enter once more upon this well-beaten track.

It is a fact worthy of note that, although numerous other alkaline copper solutions have been proposed from time to time for the determination of reducing sugars, that of Fehling has been found by the majority of workers to be the most satisfactory ; and to this day the prescription of Fehling, with but few modifications, is adhered to by most chemists for the preparation of the alkaline copper solution.

The methods of determination of sugars gravimetrically by the cupric reduction method leave little to be desired on the score of accuracy when applied to comparatively pure sugars. Even here, however, there are possible sources of error. If a paper filter be employed for collecting the cuprous oxide, it is not possible to remove the whole of the unreduced copper (from the excess of Fehling's solution employed) from the filter-paper, and, unfortunately, the quantity of copper so retained is far from constant. Again, if a Soxhlet asbestos filter or a Gooch crucible be employed, it is exceedingly difficult to wash out the last traces of alkali from the precipitated cuprous oxide. In any case it is necessary in exact work to make a blank experiment with every set of determinations.

Commercial sugars and malt worts, however, contain salts of alkali earths, nitrogenous organic matters, and other substances capable of vitiating the accuracy of the results. Certain alkali earth salts may, under the conditions of the experiment, be precipitated together with the cuprous oxide, whilst nitrogenous organic matters may dissolve a portion of the cuprous oxide. Errors of the first kind may be avoided by dissolving the moist cuprous oxide after it has been washed, and determining the copper in the solution volumetrically. One of the best methods of effecting this object is to dissolve the cuprous oxide in an acidified solution containing an excess of iron alum, and then titrate the resulting ferrous salt with $\frac{N}{10}$ permanganate. This and similar methods do not, however, eliminate the error due to the solvent action of nitrogenous organic matter.

The determination of sugars by direct titration with Fehling's solution is, on the other hand, but a rough-and-ready process, except when pure sugars are being dealt with. With commercial products giving dark-coloured solutions and containing nitrogenous organic matters, it is not possible to obtain accurate results, even when potassium ferrocyanide is used to indicate the point at which the precipitation of the cuprous oxide is complete. It was this fact which led F. W. Pavy several years ago to devise an ammoniacal copper solution for use with animal products. By means of this solution cupric reduction takes place without the precipitation of cuprous oxide, the latter being dissolved by the ammonia; the final point is determined by the disappearance of the blue colour. Those who have used Pavy's solution know, however, its many disadvantages—the inconvenience of working with an ammoniacal solution, and the much greater dilution of the liquid as compared with the ordinary Fehling's solution. Pavy's method is, in fact, only applicable for the determination of amounts of sugars from, say, 1 to 10 per cent., the errors involved being too great to enable it to be used for accurately determining larger amounts of sugars.

The titration of raw sugars, malt worts, and other commercial products with Fehling's solution, employing potassium ferrocyanide as indicator, is, as already stated, anything but an accurate method. It is also a very tedious one; and when certain amino compounds are present in the solution being titrated, so much cuprous oxide may be dissolved that it is impossible to obtain an acidified filtrate which gives no colour with potassium ferrocyanide. This arises from the fact that, almost immediately a solution of cuprous oxide is acidified, it is oxidized to cupric salt. What, therefore, is needed to increase the accuracy, and at the same time expedite the method, is an indicator which responds to a minute trace of cupric salt, and can be employed without filtering off a portion of the assay liquid, which is necessary when potassium ferrocyanide is used.

Several indicators which can be used in the manner just mentioned for determining the end-point in titrating with Fehling's solution have been proposed, and among these may be mentioned that recently suggested by E. F. Harrison (*Pharm. Journ.*, 1903, lxxi., 170), which consists of a solution of starch and potassium iodide. When a small quantity of this solution is acidified with acetic acid and brought in contact with a cupric salt, liberation of iodine occurs, and the well-known blue colour which iodine gives with starch is developed.

The indicator we have devised, and which is certainly the most satisfactory of any we are acquainted with for titrating sugars with Fehling's solution, is a solution of ferrous thiocyanate. When a drop of this indicator is placed on a slab, and a drop of a solution containing cupric salt brought in contact with it, oxidation of the ferrous salt occurs, with the immediate production of the well-known red colour of ferric thiocyanate.

In order that the indicator may possess the greatest possible delicacy, it appears to be necessary to follow certain conditions in preparing it, and our experience shows that the following prescription is the most satisfactory. One gram of ferrous ammonium sulphate and the same quantity of ammonium thiocyanate are dissolved in 10 c.c. of water at a moderate temperature—say between 45° and 50° C.—and immediately cooled; 50 c.c. of concentrated hydrochloric acid are then added. The solution so obtained has invariably a brownish-red colour, due to the presence of ferric salt, which latter must therefore be reduced. For this purpose we have found zinc dust the most satisfactory reagent to employ, and as a rule a mere trace suffices to decolorize the solution. The indicator when kept for some hours develops the red coloration by atmospheric oxidation. It may, however, be decolorized by the addition of a further quantity of zinc dust, but its delicacy is impaired after it has been decolorized several times. Prepared according to these instructions, the limit of sensitiveness of the indicator is, at least, 2 c.c. of Fehling's solution diluted to 1 litre. It should be stated that less copper than is contained in 2 c.c. of Fehling's solution diluted to 1 litre can be detected by potassium ferrocyanide, not, however, in presence of the organic matters which occur in the Fehling's titration liquid; and, again, the potassium ferrocyanide indicator requires the filtration of the titration liquid.

It is usual in sugar titrations to dilute the Fehling's solution with an equal volume of water. When working with our new indicator, it is preferable, however, not to dilute the Fehling's solution, and all the experiments herein quoted have been carried out in this manner. This procedure has the obvious advantage, other things being equal, that the maximum sharpness of the end-point in the titration is insured.

We prepare the Fehling's solution in the following manner:

Solution No. 1.—69·278 grams of crystallized copper sulphate are dissolved in water, and the solution made up to 1 litre.

Solution No. 2.—346 grams of crystallized Rochelle salt are dissolved in hot water, mixed with 142 grams of caustic soda, also dissolved in water, and after cooling made up to 1 litre.

Equal volumes of these two solutions are accurately measured out at 15·5° C. and mixed in a dry flask. The mixture constitutes the solution from which measured quantities are taken for titration. The mixture should be prepared daily.

One great advantage which the volumetric method possesses over the gravimetric is that the operator is independent of tables and results obtained under conditions not necessarily identical with those under which the analysis is being conducted. In the volumetric method, each separate preparation of Fehling's solution may be standardized under the exact conditions employed in any given analysis.

The method of titration is as follows: Freshly mixed Fehling's solution (10 c.c.)* is accurately measured into a 200 c.c. boiling flask and raised to boiling. The sugar solution, which should be adjusted to such a strength that 20 to 30 c.c. of it are required to reduce 10 c.c. of Fehling's solution, is then run into the boiling liquid in small amounts, commencing with 5 c.c. After each addition of sugar solution the mixture is boiled, the liquid being kept rotated. About a dozen drops of the indicator are placed on a porcelain or opal glass slab, and when it is judged that the precipitation of cuprous oxide is complete, a drop of the liquid is withdrawn by a clean glass rod or by a capillary tube, and brought in contact with a drop of the indicator on the slab. The test must be carried out rapidly. It is also essential to perform the titration as rapidly as possible, as an atmosphere of steam is then kept in the neck of the flask, and the influence of atmospheric oxygen avoided. At the final point the liquid is boiled for about ten seconds. As in the ordinary volumetric method, the first titration may only give approximate results, and a second or third will then be necessary to establish the end-point accurately. However, when the operator has gained experience, the first titration is as much to be relied on as succeeding ones, and this point is clearly brought out in the results we shall cite. One titration takes from two and a half to three minutes.

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* In some cases it is advisable to employ 20 c.c. of Fehling's solution in the titration.

The following are a few examples of results obtained with the method:

Invert Sugar, Series A.—Pure sucrose (0·95 gram) was dissolved in water (150 c.c.), and boiled with $\frac{N}{2}$ hydrochloric acid (30 c.c.), the mixture being maintained in ebullition for one minute, cooled, neutralized by the addition of $\frac{N}{2}$ sodium hydroxide (30 c.c.), and made up with water to 500 c.c. This solution, which contained 0·2 gram of invert sugar per 100 c.c., was titrated with 10 c.c. portions of Fehling's solution prepared as above described, with the following results:

Fehling's Solution.	Invert Sugar Solution.	Weight of Invert Sugar corresponding with 1 c.c. of Fehling's Solution.
1. 10 c.c.	25·3 c.c.	0·00506 gram.
2. „	25·4 „	0·00508 „
3. „	25·5 „	0·00510 „
4. „	25·6 „	0·00512 „
5. „	25·2 „	0·00504 „
6. „	25·1 „	0·00502 „

Mean of six titrations: 1 c.c. of the Fehling's solution corresponds with 0·005066 gram of invert sugar.

Invert Sugar, Series B.—The invert sugar solution employed in these titrations was prepared in the same manner as that used in Series A; another preparation of Fehling's solution was used.

Fehling's Solution.	Invert Sugar Solution.	Weight of Invert Sugar corresponding with 1 c.c. of Fehling's Solution.
1. 10 c.c.	25·4 c.c.	0·00508 gram.
2. „	25·6	0·00512 „
3. „	25·6	0·00512 „
4. „	25·4	0·00508 „
5. „	25·4	0·00508 „

Mean of five titrations: 1 c.c. of the Fehling's solution corresponds with 0·005096 gram of invert sugar.

Dextrose.—The specimen of this sugar was prepared from invert sugar and repeatedly recrystallized from purified wood-spirit.[*] As received, it contained a trace of spirit. An aqueous solution was prepared and evaporated, the residue taken up with water and again evaporated. This was repeated until the whole of the spirit was removed. The final solution had a specific gravity at 15·5°/15·5° of 1018·93. Applying the solution divisor of Brown, Morris and Millar, the concentration would therefore be 4·929 grams per 100 c.c. (fluid grams). It read 5·20° in the polarimeter. The specific rotatory power was therefore $[\alpha]_D$ 52·76°.

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[*] We are indebted to Mr. Lewis Eynon for this specimen of dextrose. Mr. Eynon determined the specific rotatory power of the sample, and found it to be $[\alpha]_D$ 52·45° in a solution containing 7·88 grams in 100 c.c.

A portion of 25 grams of this solution was carefully weighed out and diluted with water to 500 c.c. This 25 grams is equal to 24·535 fluid grams, and contained 1·2093 grams of the dextrose.

The dilute solution employed for the titrations contained, therefore, 0·2419 gram of dextrose per 100 c.c.

The Fehling's solution was the same as used in the invert sugar experiments (Series B).

Fehling's Solution.	Dextrose Solution.	Weight of Dextrose corresponding with 1 c.c. of Fehling's Solution.
1. 10 c.c.	20·4 c.c.	0·004934 gram.
2.	20·3 ,,	0·004901 ,,
3.	20·1 ,,	0·004862 ,,
4.	20·4 ,,	0·004934 ,,
5.	20·1 ,,	0·004862 ,,
6.	20·5 ,,	0·004959 ,,
7.	20·3 ,,	0·004901 ,,
8.	20·3 ,,	0·004901 ,,

Mean of eight titrations: 1 c.c. of the Fehling's solution corresponds with 0·004907 gram of dextrose.

Maltose.—The specimen of this sugar was prepared by the action of diastase on potato starch paste. Although it was recrystallized repeatedly from alcohol, it appears not to have been quite pure. The alcohol adhering to the crystals was eliminated in the manner described under dextrose, and the specific gravity of the aqueous solution having been taken, its concentration was determined by the aid of a factor.

The specific gravity of the solution at 15·5° compared with water at the same temperature was 1036·94, which corresponds with a concentration of $c_{3\cdot93} = 9\cdot399$. The solution gave a reading in the polarimeter of 26·4° in a 200 millimetre tube, whence $[\alpha]_D = 140\cdot4°$. This somewhat high result was confirmed by a second determination; it points to the presence of a trace of one of the malto-dextrins in the maltose. The specimen may also have contained a trace of dextrose. It is exceedingly difficult to purify maltose which has been prepared by the action of malt-diastase on starch paste, especially if the reaction has been allowed to proceed near its final point. Malto-dextrins are invariably formed which are soluble in alcohol, and to remove them from the maltose, by crystallization from alcohol, is by no means easy. A much simpler and more satisfactory method of preparing maltose is that devised by J. L. Baker, namely, by the action of ungerminated barley on starch paste. This author has shown that the dextrins formed in this way are practically insoluble in alcohol, and the crude maltose is readily separated from the small amount of dextrose it contains by crystallization from alcohol.

Ten c.c. of the solution of maltose ($c_{3\cdot93} = 9\cdot399$) were diluted with water to 250 c.c., which dilute solution, containing 0·3760 gram of anhydrous maltose per 100 c.c., was titrated with Fehling's solution. The Fehling's solution was that employed in the invert sugar experiment (Series A).

Fehling's Solution.	Maltose Solution.	Weight of Maltose corresponding to 1 c.c. of Fehling's Solution.
1. 10 c.c.	21·6 c.c.	0·008121
2. ,,	21·8 ,,	0·008196

Mean of two titrations: 1 c.c. of the Fehling's solution corresponds with 0·008158 gram of anhydrous maltose.

As a result of considerable experience, we have found that under the conditions above described the cupric reducing power of invert sugar to that of anhydrous maltose stands in the ratio of 100 : 62. The maltose value of the Fehling's solution calculated from the results obtained by titrating with invert sugar is, therefore: $\frac{0{\cdot}005066 \times 100}{62} = 0{\cdot}008170$, a number agreeing fairly well with that found by directly titrating the Fehling's solution with maltose.

In the following examples of determinations with commercial products, 10 c.c. of Fehling's solution were used in each instance.

CANE MOLASSES, ONE PER CENT. SOLUTION.

	Molasses Solution.	Sugar calculated as Invert Sugar in the Sample.
1.	23·6 c.c.	21·48 per cent.
2.	23·4 ,,	21·67 ,,

CANE MOLASSES, ONE PER CENT. SOLUTION.

1.	24·9 c.c.	20·37 per cent.
2.	25·0 ,,	20·08 ,,

SAME MOLASSES AFTER INVERSION, 0·25 PER CENT. SOLUTION.

1.	33·2 c.c.	61·08 per cent.
2.	33·3 ,,	60·88 ,,

RAW CANE SUGAR, 0·5 PER CENT. SOLUTION.

1.	27·4 c.c.	37·00 per cent.
2.	27·5 ,,	36·86 ,,
3.	27·3 ,,	37·14 ,,
4.	27·5 ,,	36·86 ,,

SAMPLE OF STOUT. ORIGINAL GRAVITY 1075.

50 c.c. evaporated to remove alcohol, and made up to 200 c.c.

		Reducing Power expressed as Percentage of Maltose on Original Wort Solids.
1.	30·8 c.c.	5·64 per cent.
2.	30·7 ,,	5·66
3.	30·6	5·68
4.	30·6	5·68
5.	30·7	5·66
6.	30·5	5·70

Many other examples might have been given—*e.g.*, determinations of reducing sugars in malt wort and in caramels. Just as concordant results can be obtained with these as with molasses and stout. The volumetric determination of reducing sugars by the method we have described requires some practice in order to insure satisfactory results, but, as the above examples show, the method is capable of a high degree of accuracy, and we believe it will be found superior in all respects to the gravimetric process.

We are indebted to Mr. J. J. Belton for carrying out some of the experimental work contained in this paper.

DISCUSSION.

The CHAIRMAN (Mr. Blount), in inviting discussion, remarked that the fact that the titration could be carried out by artificial light was a very practical advantage. He should like to ask whether the authors had found any particular form of artificial light to be specially suitable.

Dr. THORNE said that he had not yet tried this process, but, from what he had just heard, he certainly thought that it would be of great assistance to those interested in the estimation of sugar. Up to the present volumetric processes had not been satisfactory, and those who had to do with sugar estimations had been compelled reluctantly to fall back on the gravimetric method. He said "reluctantly," because, much as that method had been improved by the work of O'Sullivan, Brown and Morris, and others, it still was a decidedly unsatisfactory method. So much depended upon minute details of manipulation, and upon the other constituents of more or less unknown character that might be present in commercial products. Mr. Ling had been kind enough to examine for him a sample of raw cane-sugar which had yielded somewhat uncertain results by the gravimetric method, and in that case the volumetric results came out very well, indicating that the discrepancies which occurred in some of the other volumetric processes had been—at any rate to a great extent—obviated.

Mr. BAKER said that a short time ago Mr. Ling had outlined this process to him, and it had been tried in his laboratory with results the satisfactory character of which Mr. Dick, who had carried out the determinations, would be able to confirm. The new indicator had been tried side by side with the ordinary ferrocyanide indicator with great success. It had a very suitable application in the determination of the diastatic power of malt, according to Mr. Ling's modification of Lintner's directions. Following that modified method, and using this indicator, it was possible to save, on a dozen determinations of diastatic power, at least ten minutes—a very important saving when the number of analyses was large. He had found the process to work very well by electric light.

Mr. W. D. DICK said that he had used the authors' process in Mr. Baker's laboratory side by side with the filtration process for the last two months, and had found it to be considerably quicker and more satisfactory in every way.

Mr. E. H. JEFFERS congratulated the authors on their historical sketch of the volumetric methods of estimating sugar. In the process now described there were one or two points in manipulation which seemed to him rather risky. In the first place, the quantity of Fehling's solution used—namely, 10 c.c.—seemed very small. In working gravimetrically it was usual to use 50 c.c., and, even under conditions conducing to the greatest accuracy, it was almost impossible to get duplicate results to agree within less than a milligram, which on 50 c.c. of Fehling's solution meant an experimental error of about ½ per cent. If smaller quantities were worked upon, the error was correspondingly multiplied. He quite agreed that the gravimetric method was not free from liability to error. In the first place, a blank experiment was necessary in every determination, but it was quite uncertain whether the result of the blank experiment could be applied as it stood. For instance, the filter-paper could never be washed perfectly free from the blue salt which it absorbed, and which had been shown to be an alkaline salt. He had found, however, that, while the filter-paper could be washed until the washings were perfectly free from alkali, the paper itself remained alkaline, which he thought must be due to the blue salt. In making an estimation on 25 c.c. of a 1 per cent. sugar solution, all, or very nearly all, of the blue salt was decomposed, and consequently the quantity absorbed by the filter-paper was much less than in the blank experiment. With a view to getting over this difficulty, he had tried the method mentioned in the paper, of dissolving off the copper oxide with iron alum and titrating with permanganate, and had found that to give very accurate results.

Mr. CHAPMAN said that the great difficulty in getting accurate results with Fehling's solution was that of keeping the conditions as to dilution, proportion of sugar to copper, etc., perfectly constant. It seemed to him that this might be a matter of greater difficulty in a volumetric than in a gravimetric method, though, of course, it could be to a great extent got over by adjusting the strength of the solutions employed. He noticed that the relationship between the reducing power of invert sugar and that of dextrose appeared to be very different in the volumetric and in the ordinary gravimetric method. In the gravimetric method, with the usual dilution, it was about 97 for invert sugar (dextrose = 100), whereas according to the figures which the authors had given for the volumetric method it was distinctly less, so that the reducing conditions must be quite different in the two cases. He would like to ask whether, in the case of very dark molasses, the colour of the solution after filtering interfered in any way with the delicacy of the production of the red coloration with the indicator.

Mr. E. R. BOLTON asked whether the authors had ever compared their process with another gravimetric process, which he thought was due to Soxhlet, in which the cuprous oxide was filtered in an asbestos filter-tube, washed, and reduced in hydrogen. That method in his hands had given very good results, but his experience with it was not large.

Dr. SCHIDROWITZ said that he should like to hear again how the Fehling's solution was standardized, as he had not quite followed this. He, in common with many others, invariably used the Soxhlet method, filtering through asbestos. In that method all trouble with blank experiments was overcome.

The CHAIRMAN said that he had occasion, in another branch of analytical work, frequently to measure small quantities of liquids with great accuracy, and he should never think of making those measurements by the simple observation of a pipette or any calibrated vessel—he weighed the liquid; and he would suggest that possibly some of the objections which had been urged against the authors' process might be based upon this fundamental source of error, which was common to all volumetric determinations.

Mr. LING, in reply, said very good results could be obtained by working with ordinary incandescent gas light. The results given in the paper showed the accuracy of the volumetric process, and the use of 10 c.c. of Fehling's solution instead of 50 c.c. as in the gravimetric method had no bearing on the relative accuracy of the two methods. He had had a large experience of the gravimetric method, and had hitherto used it for research work, because there were with pure solutions no disturbing factors, and concordance of results might safely be taken as indicating accuracy.

Dr. Dyer asked whether, in the case of the black molasses, a solution of the original material was worked upon, or whether it was first clarified with lead acetate.

Mr. Ling said that the original material was worked upon, without clarification, all reducing bodies being taken as sugar, which, strictly speaking, was not the case. He had for many years used the method referred to by Mr. Bolton. The Fehling's solution was standardized, as described in the paper, by invert sugar or by any other pure reducing sugar. The objection attaching to the use of calibrated vessels applied, of course, to this as to other volumetric processes. One of the difficulties of this process was that very dilute solutions must be used, so that any errors were largely multiplied. The errors, however, could be reduced to a minimum by careful working, especially if the dilutions were made by weight.

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THE DETERMINATION OF HIGHER ALCOHOLS IN SPIRITS.—I.

By PHILIP SCHIDROWITZ, Ph.D., and FREDERICK KAYE, A.R.C.Sc.

(Read at the Meeting, April 5, 1905.)

Introductory.

The present experiments were undertaken with a view to examining the relative accuracy, as far as possible, of the various processes devised for the determination of the higher alcohols in spirits, and not primarily with the object of putting forward any new method of our own. The necessity for some revision of this kind is so obvious to all chemists working in this branch of applied analytical chemistry, that an apology for its publication is scarcely necessary; but we may say, in explanation of the apparently scanty nature of the present communication, that the difficulties of the work have exceeded our anticipations. The experiments were commenced some eighteen months ago, but owing to unavoidable interruptions, and to the difficulties encountered, we are not yet in a position to publish the whole of the results obtained so far, as many of these require further confirmation. We nevertheless think it desirable to make this preliminary communication now, stating the general scope of the work, the methods of purification of the substances employed, and giving one short but, we believe, important series of results, namely, those concerning the Beckmann process, of which we, in common with many others, had hoped much, but which, unfortunately, was found to give entirely unsatisfactory results. As far as we are aware, this is the first criticism of this process that has appeared, and this fact—as it may save other workers much time and trouble—we venture to put forward in extenuation of what might otherwise be regarded as a somewhat premature paper.

Scope of Work.—We had as our object the examination of such processes only as seemed *a priori* to afford a reasonable probability of success. We selected the Beckmann nitrite process (E. Beckmann, *Zeit. Unters. Nahr. u. Genussm.*, ii., 709, and ditto, iv., 1057), the Allen-Marquardt method (ANALYST, 1891), as modified by one of us (Schidrowitz, *Journ. Soc. Chem. Ind.*, June, 1902), and finally the French colorimetric process (Girard et Cuniasse, " Manuel pratique de l'analyse des alcools et des spiritueux," Paris, 1899 : Masson). We purposely refrained from further experiments with the German official process (Röse-Stutzer-Windisch, *Arbeit des Kaiserl. Gesundheitsa.*, 1889, v., 391), inasmuch as one of us (Schidrowitz, *loc. cit.*) had shown that for one class of spirit, namely, whisky, this process was, at any rate, quite out of the question ; it gave, in fact, negative results. As it appeared likely that this might also apply to other spirits, and as one of us had had private communications from distinguished colleagues in several parts of the world confirming these observations, we did not think it worth while to devote our time to the process.

Purification of Materials ; Ethylic Alcohol.—The "pure absolute alcohol" of commerce was subjected to fractional distillation in a flask provided with a Young's "rod and disc" head until the resultant product showed no aldehyde reaction, was entirely neutral, and gave satisfactory blank tests with both the Allen-Marquardt and the colorimetric processes.

Carbon Tetrachloride.—This was first shaken with water, then boiled for several hours with chromic acid mixture, subsequently washed first with water, then with very dilute sodium bicarbonate, and finally with distilled water until neutral. The product so obtained was then further purified by distillation.

Amylic Alcohol.—Several methods of purification of the "pure" amylic alcohol of commerce (B.P., 128-132) were tried, but the following was finally adopted as being most satisfactory. The alcohol was successively shaken with dilute sulphuric acid, water, warm milk of lime, then filtered and allowed to stand overnight over solid quicklime, distilled over the latter, treated with freshly ignited sodium sulphate, distilled over the same, and finally fractionated by means of a Young's "rod and disc" apparatus. By this means we were able to obtain a product which distilled entirely within half a degree, namely, at 129·5 to 130° C.

Apparatus.—We found it a matter of very considerable difficulty to obtain an apparatus for the prolonged boiling, in the Allen-Marquardt process, of the carbon tetrachloride solution with the chromic acid mixture which was absolutely air-tight, and would permit of prolonged boiling without loss. Rubber cannot be used, and ordinary corks were found very unsatisfactory. In the same way, neither an ordinary (24-inch) Liebig nor a five-bulb condenser was capable of preventing all loss. Finally we were compelled to resort to an all-glass apparatus, consisting of a 300 c.c. Jena glass Erlenmeyer flask very carefully ground in to a 24-inch Liebig condenser, the central tube of the latter being fitted throughout its length with a Young's "rod and disc." This effectually prevents the slightest loss, and our experience is that an apparatus of this kind, if properly made, lasts for a very considerable time without cracking, and is useful for many other purposes in the laboratory, notably for the saponification of the ethers in spirits. Otherwise the apparatus used needs no special description.

A. The Beckmann Process.

This process consists substantially in a separation of the higher alcohols from the spirit (to which a certain amount of calcium chloride has been previously added) by means of carbon tetrachloride, converting the higher alcohols so isolated into their nitrites by means of nitrous acid set free by the interaction of sodium nitrite and sodium bisulphate, removing the excess of nitrous acid with sodium bicarbonate, decomposing the nitrites with sulphuric acid, and finally titrating the nitrous acid so obtained with permanganate.

Our first experiment was made (following Beckmann's instructions minutely in every detail) with a blank solution of 48·7 per cent. alcohol (by volume). The final titration in this case required some 300 c.c. $\frac{N}{30}$ permanganate, corresponding roughly to 0·8 per cent. of amylic alcohol. This was obviously an impossible result, but blank tests made with the materials (carbon tetrachloride, ice, sulphuric acid, etc.) employed failed to yield any explanation. It became therefore necessary, in the first place, to ascertain whether the fault lay in the actual process of extraction or in the treatment of the carbon tetrachloride solution with nitrous acid, etc. In order to test this point, 150 c.c. of pure carbon tetrachloride were treated with sodium bisulphate and sodium nitrite, as described by Beckmann, were then filtered, the residue being washed in accordance with the instructions, the filtrate treated with bicarbonate, water added until the excess of salt was dissolved, and the whole of the carbon tetrachloride then divided into two portions, one consisting of two-thirds of the whole, the other of the remaining third. The former was finished off according to Beckmann, and required 1·0 to 1·5 c.c. $\frac{N}{30}$ permanganate, the end, point not being sharply marked. The latter was washed with small quantities of water, filtered through a dry filter, the filter washed with a little tetrachloride, and the filtrate then treated in the manner described. In this case 0·5 c.c. permanganate was required. From these results it was quite clear that the process of nitration and subsequent removal of excess of nitrous acid, etc., is not at fault. A second blank experiment with ethylic alcohol only was made, with results practically identical with the first one. It was fairly obvious, therefore, that the extraction was the weak point, and that under the conditions employed by us a certain amount of ethylic alcohol was extracted by the tetrachloride. As we followed Beckmann's instructions minutely, we could only surmise that Beckmann in his experiments employed some peculiarly favourable alcoholic strength; for in this respect his instructions are very vague, saying as he does that the strength of the spirit to be extracted is not to be more than 50 per cent., but indicating no particular strength, as one might have expected. We therefore attempted to modify the method of extraction, finishing with the usual nitration. It struck us that in this connection the Allen-Marquardt method of extraction, which requires an approximately (according to Allen and Chattaway, *loc. cit.*) exact specific gravity of 1·1 for the solution of brine and alcohol from which the higher alcohols are extracted, might prove useful, and some experiments in this direction were therefore made; these were as follows:

1. One hundred c.c. of spirit (strength as above) were diluted with brine until they showed a specific gravity of 1·1 (by the hydrometer), and were then extracted

according to Allen. The final tetrachloride solution was then treated as in the Beckmann process. Forty c.c. permanganate were used, corresponding in terms of amylic alcohol to 1·1 grams per litre.

2. Two hundred c.c. were extracted with tetrachloride, according to the Allen method, at 15° C. exactly, and at an exact specific gravity of 1·10. After extraction the carbon tetrachloride was divided into two equal portions, the one being treated according to Beckmann, the other according to Allen.

The first required 32 c.c. permanganate (= to, roughly, 0·7 gramme per litre); the second, 2·8 c.c. $\frac{N}{10}$ baryta, of which a part was ascribable to mineral acid. If, however, in this particular case the whole of the 2·8 c.c. baryta employed were calculated in terms of amylic alcohol, it would amount to 0·24 gramme per litre, or about one-third of the blank found in the corresponding Beckmann experiment. As stated, however, a part of the Allen blank was due to mineral acid.

3. Repeated Experiment 2. In this case the Beckmann portion required 31·5 c.c. permanganate (= to, roughly, 0·7 gram per litre); the Allen portion required 2·8 c.c. $\frac{N}{10}$ baryta, of which one half was due to mineral acid. The blank in this case was, therefore, 0·12 gram per litre.

4. As Experiment 3, except that the specific gravity of the spirit extracted was purposely raised to 1·1235. In this case the Beckmann portion required 25 c.c. permanganate (= to, roughly, 0·4 gram per litre); the Allen portion, as before, an amount of baryta equivalent to, roughly, 0·1 gram per litre.

5. As before, but specific gravity of the fluid extracted was 1·0938. In this case the Beckmann portion required 35 c.c. permanganate (= to, roughly, 0·95 gram per litre); the Allen portion required (excluding the mineral acid) 3·8 c.c. baryta—that is, an amount equivalent to, roughly, 0·34 gram per litre.

The latter experiment indicates that in the Allen-Marquardt process it is desirable not to allow the specific gravity of the alcoholic brine to fall below 1·10.

From these experiments we conclude:

1. That the Beckmann process as published is unworkable.

2. That the fault lies not in the latter (nitration) part of the process, but in the actual extraction.

3. That the Allen-Marquardt process of extraction undoubtedly does ultimately leave a certain quantity of ethylic alcohol in the carbon tetrachloride extract, but that for some reason unexplained the greater part of this disappears during the oxidation with chromic acid mixture, but is not converted into acetic acid. For this curious phenomenon, which may perhaps account for the somewhat anomalous results occasionally obtained by this process, we suggest the following in explanation:

(*a*) The alcohol is split up into carbonic acid and water; or (*b*) the alcohol forms, by the action of mineral acid present or produced, an organic compound, such as ethyl chloride, for example, which does not possess acid properties.

Whether this curious action affects the higher alcohols also we are not yet in a position to say, but we hope in our second paper to be able to throw some light on this point in the course of our consideration of the Allen-Marquardt process as a whole. We are still inclined to believe that, if carefully worked, this process is still the most reliable of all those published. It appears to be very little known in some Continental quarters, particularly where the Röse process is most in favour, and in this connection we think it necessary to draw attention to some recent remarks by Karl Windisch on the subject. In a paper on Brandy contributed to the *Zeit. Unters. Nahr. u. Genussm.* (No. 8, October 15, 1904), he says (p. 488): "Ph. Schidrowitz examined twelve commercial whiskies, and determined the fusel-oil by the Alien-Marquardt method. In this process the fusel-oil is shaken out with *chloroform,** etc. . . . This process gives *results which are much too low*" (*cf.* Karl Windisch, *Arbeit des Kaiserl. Gesundheitsa.*, 1889, v., 373). Now, this statement clearly indicates that Windisch is not familiar with the Allen modification of the Marquardt process, for one of the improvements due to Allen was the substitution of carbon tetrachloride for chloroform. It is also somewhat unfortunate for the contention of Windisch that the work to which he refers as having disproved the value of the Allen-Marquardt process was published some two years before the Allen-Marquardt process saw the light. His criticism, therefore, of the work of one of us in this respect is, to say the least, somewhat premature.

DISCUSSION.

The PRESIDENT (Mr. Bevan), in inviting discussion, said that it seemed obviously very important, not only that the alcoholic strength should be well maintained, but also that the strength of the brine solution should be kept as constant as possible. It would probably be satisfactory if a rule could be laid down for this, and he hoped that the authors might find it possible to do so in the second contribution to the knowledge of this subject which he understood they would make later on.

Mr. DIBDIN inquired whether the authors had found the temperature to vary as the strength of the solution varied, and if so, whether they had made any attempt to keep it constant.

Dr. SCHIDROWITZ said that they had all through tried to keep the temperature of the various solutions during shaking out at as nearly as possible 15° C.

Dr. J. T. HEWITT said that the authors seemed to have experimented with every available process for the determination of higher alcohols, and also to have judged the results in such a way that they knew pretty well what was to be expected from any given mode of procedure. That could not be said in every case. For instance, in the official German method—the Röse-Hertzeld method—which consisted in shaking out and merely measuring an increase in volume, it was difficult to see how any exact idea could be obtained of what it was that was being measured in any particular case. With regard to the reflux apparatus used by the authors, he had recently had the privilege of seeing this in use in Dr. Schidrowitz's laboratory, and there could be no doubt that it worked most admirably. The efficiency of condensation due to the use of the rod and disc column was surprising. It was interesting to know that a combination of the Beckmann and Allen-Marquardt methods gave good results, for both these methods seemed to be based on fairly sound principles. He should have thought that the colorimetric method would have proved more satisfactory than appeared to have been the case. Dr. Schidrowitz, however, he believed, considered that there were a good many other substances that might affect the coloration obtained with strong sulphuric acid. He (Dr. Hewitt) had been at one time inclined to think that not merely the higher alcohols, but also the higher acids, might have some influence, and he had tried removing the ethers, in addition to other by-products, by prolonged boiling with caustic potash, and then determining the higher alcohols colorimetrically; and he had been surprised to find that the result was not very different from that obtained when merely the aldehydes and furfural were removed before distillation. Dr. Schidrowitz, he thought, held the view that the other substances in question were not of the nature of ethers or acids, but were of an entirely different type; and he (Dr. Hewitt) was now inclined to agree in this.

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* The italics are ours.

Mr. HERBERT E. BURGESS thought that it would be of much assistance to other workers if full details were given of the apparatus which the authors had used, and particularly of the still-head. This was different from any he had seen before. The still-head of Dr. Young was, of course, familiar, but he had never seen one with a condenser outside it. He could confirm the authors' views as to rubber connections. He had had considerable experience in distillation, and had found that, where rubber could not be used, a perfectly tight joint could be made with a wooden stopper painted round with ordinary mucilage and covered with a piece of paper, as recently described by Mr. Page in the *Chemical News*. He had been wondering whether a satisfactory estimation of the higher alcohols could be made on the lines of the method described by Dr. Thorpe for the estimation of ethers in alcoholic solution, namely, by distilling a definite quantity with a still-head of the Young-Thomas type, shaking out with petroleum ether, acetylating and saponifying.

The PRESIDENT said that he had rather expected some mention to be made in the paper of a new apparatus made by Messrs. Priem for the purpose of getting over the difficulty due to corks. The flask was made with a glass trough round the outside of the neck, and a mercury seal was formed by slipping over the neck of the flask another tube which dipped into mercury contained in the trough. Its only disadvantage was its liability to break, and, with a view of obviating this, he (the President) had suggested that the mercury should be retained by means of an india-rubber stopper fixed on the neck of the flask between it and the outer tube, which fitted closely over the outside of the stopper. This modification was simple to make, and had been found to work very satisfactorily.

Mr. CHAPMAN said that owing to the difficulties which had been experienced in connection with the estimation of higher alcohols in spirituous liquids, and to the large differences which had in many instances been recorded between the results obtained by different chemists employing different methods, it was highly important that some definite information should be obtained on this subject, and to this end it was suggested to Dr. Schidrowitz and his colleague that they should direct their attention to the matter under the provisions of the Analytical Investigation Scheme which the Council of the Society had recently initiated. These gentlemen had taken the matter up very heartily, and he (Mr. Chapman) was sure that all would agree with him that the investigation could not have been placed in better hands. He thought that this was the first occasion on which the various methods for the estimation of the higher alcohols had been submitted to a critical and comparative study, and in view of the smallness of the quantities that had to be estimated, and of the very great chemical difficulties in the way, it would easily be realized how troublesome the investigation must have been. The difficulty of obtaining perfectly pure ethylic alcohol and of the preparation of solutions containing known quantities of the higher alcohols must at the outset have been very considerable. He (Mr. Chapman) had personally had experience of three methods for the estimation of the higher alcohols in spirituous liquids. The earliest was the physical method of Traube, in which an instrument known as a stalagmometer was employed. This method was only capable of being used in certain cases, and possessed a big inherent error, and he was under the impression that it was now rarely, if ever, employed. In addition to this, he had worked with Röse's process, which, so far as his experience went, was useless, and also with that of Marquardt as modified by Allen. This last-mentioned method he had used for some years, and his experience had been, on the whole, that of the authors of the present paper—viz., that it was a useful method, and one which, if properly carried out, was capable of giving good results. A great advantage which this method possessed, moreover, was that the actual oxidation products of the higher alcohols were obtained, and could be further used for purposes of examination or identification. Next to the specific gravity of the brine liquid he thought the factor which had the greatest effect on the results was the washing of the carbon tetrachloride extract. In his own experience, he had occasionally found that abnormal and impossible results were obtained by this method even when the greatest care was employed in its execution, and he would like to ask the authors whether they had ever had a similar experience. In the cases to which he referred the organic acids produced would require perhaps three or four times as much standard alkali for neutralization as they ought to, and he had usually observed at the same time that the mineral acid, instead of requiring the customary 1 c.c. or 2 c.c., would require perhaps 6 c.c., or even more, of alkali for neutralization. He would also like to ask the authors whether they had yet made any experiments with the colorimetric method which was so largely employed in France.

Dr. Schidrowitz, in reply, said that they had a number of figures which were not included in this paper, because they considered that they required amplification. The present paper was merely a preliminary communication recording the revision and control of the Beckmann method and the establishment of certain facts with regard to the Allen-Marquardt method, which would be productive of one or two slight changes in the apparatus at present in use, and so forth. Their further results would be published later on, and would include observations on the colorimetric method, with which they had made a considerable number of experiments already. With regard to the colorimetric method, although it gave fairly good results with pure amylic alcohol or iso-butylic alcohol, it obviously could not give a satisfactory result in a spirit containing a mixture of various alcohols. It was known, for instance, that, while iso-butylic alcohol gave a very strong reaction with sulphuric acid, normal propylic alcohol gave practically no reaction, and the amylic alcohol of fermentation gave a coloration which was not as strong as that given by iso-butylic alcohol. In a fermented liquid like whisky, the higher alcohols consisted, according to Bell—whose figures were the only ones available—of a mixture of about 35 per cent. of butylic alcohol, 35 per cent. of amylic alcohol, and 30 per cent. of propylic alcohol. The proportions, however, varied in every spirit, and the results obtained by acting on such a mixture with sulphuric acid and referring the colour to a standard for one particular alcohol must necessarily be misleading. They had lately made a good many parallel estimations by the Allen-Marquardt method and by the colorimetric method, and had worked out a new curve for amylic alcohol. The results were certainly interesting. He did not, however, think that they indicated higher alcohols pure and simple, but probably a variety of substances; and he agreed with

Dr. Hewitt that these probably included terpenes and substances which were not removed by saponification with potash. They had made the experiment, which Dr. Hewitt mentioned, of boiling for a long time with potash and then comparing the colorimetric results with those obtained after removing merely the aldehydes and furfural, and they had found a difference, but scarcely a radical one. The idea of putting the disc and rod column inside the condenser was their own. He thought that Dr. Thorpe's process for estimating esters would not be suitable in this case. It was only intended to apply to spirits or liquids containing relatively much larger quantities of those bodies. A process on the same lines was used for liqueurs, but they had tried it with whisky and brandy, and had found it impracticable because the quantities were so small. With regard to cork, certain substances were extracted from it by carbon tetrachloride and ether—a fatal objection to its use when the constituents to be estimated were so very minute in quantity. Ground-glass connections seemed by far the simplest, and, if properly made, seldom cracked. They had had flasks with such connections in constant use for over a year.

The PRESIDENT inquired what was used to make the joint tight.

Dr. SCHIDROWITZ said that in the Allen-Marquardt method they used sulphuric acid, but, as a matter of fact, a well-ground connection did not need anything at all. Continuing, he said that they had found that abnormal results such as Mr. Chapman mentioned were occasionally obtained, the mineral acid in the blank determination being in such cases also too high, but that in the course of a very large number of determinations recently made they had not had trouble in this direction. They could not account for this. The washing of the carbon tetrachloride solution was always carried out in the same way, and he thought it must be due to some other cause—possibly to decomposition. The mercury seal, which the President had mentioned, was originally due to Dr. Wiley, of Washington, who, however, used a differently-shaped flask, which, though suitable for fat extraction, did not readily permit of the residue being removed from the flask for quantitative purposes. They had not found them liable to crack.

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THE ANALYSIS OF SAMPLES OF MILK REFERRED TO THE GOVERNMENT LABORATORY IN CONNECTION WITH THE SALE OF FOOD AND DRUGS ACT.

BY DR. THOMAS E. THORPE, C.B., F.R.S.

(*Reproduced in somewhat abridged form from the Journal of the Chemical Society*, 1905, lxxxvii., 206.)

THE samples of milk submitted to the Government Laboratory under the provisions of the Acts are invariably more or less sour when received, and hence it is of importance to determine whether this fact in any way prevents a true inference as to the character of the fresh milk, or interferes with the determination of the degree of sophistication to which the milk may have been subjected.

It would appear that bacteria which produce steatolytic enzymes do not develop in sour milk to any considerable extent, since the proportion of fat suffers little, if any, alteration during the souring of the milk. Thus, of thirteen samples of genuine milk, the fat in which was determined when fresh and after keeping for periods varying from two to fourteen and a half weeks, the largest decrease in fat percentage observed was 0·15 (after thirteen weeks), while the average decrease was only 0·06 per cent. The presence of added water does not affect these results, which show that if the fat in milk suffers any alteration in amount as the milk becomes sour, it is too inconsiderable to affect to any substantial extent the experimental proof of the validity of any charge based on an alleged deficiency of fat.

The proportion of non-fatty solids in milk is, however, undoubtedly altered to a greater or less extent by the fermentative changes associated with its souring. As is well known, the principal constituent of milk suffering such change in the process of souring is the lactose. Lactic acid is produced at an early stage, the rapidity of its formation depending, within limits, upon the temperature. This reaction is produced by micro-organisms, of which there are over 100 kinds capable of effecting the change. It should be noted that in the usual way of expressing the reaction—

$$C_{12}H_{22}O_{11},H_2O = \underset{\text{(dextrose)}}{C_6H_{12}O_6} + \underset{\text{(galactose)}}{C_6H_{12}O_6}, \text{ and } \underset{\text{(galactose)}}{C_6H_{12}O_6} = 2CH_3.CH(OH).CO_2H$$

no loss of weight is involved. Moreover, the lactose is never wholly transformed under ordinary conditions into lactic acid and dextrose, generally more than half remaining unaffected after a lapse of about eight weeks, although in some exceptional cases about 60 per cent. has suffered conversion. The activity of the organisms concerned in this change appears to be inhibited when the lactic acid reaches a certain limit.

If this were the only change taking place in the souring of milk, the neutralization of the lactic acid by a known weight of strontia and the determination of the total weight of milk solids, less the corresponding weight of strontia added, would give the equivalent of the solids in the fresh milk. As a fact, however, other changes actually do take place, and these involve a slight loss of weight. Thus, a series of fifteen experiments showed that the loss in non-fatty solids during two to fourteen and a half weeks ranged from 0·24 to 0·87 per cent. for genuine milks, while in the case of watered milks the loss amounted to 0·23 to 0·68 per cent. Similar results were obtained by the examination of separated and watered separated milks, the loss in the latter cases amounting, after a lapse of fifty-five and fifty-seven weeks, to about 1·45 per cent. of non-fatty solids. Thus it is clear that, concurrently with the formation of lactic acid, there are produced substances which are either gaseous at ordinary temperatures, or are volatilized during the operation of determining the solids of the milk. Although not large in amount, this loss of weight is sufficient to affect any estimation of the degree of sophistication to which the milk may have been subjected.

Butyric acid is a product of the early stages of the souring of milk, although the proportion in which it may be formed is probably very small. It is certain that milk in which the butyric stage of fermentation has become very pronounced is of relatively infrequent occurrence in the samples received for analysis at the Government Laboratory. Acetic acid is, however, almost invariably present in these samples, but the proportion rarely exceeds 0·2 per cent. at the end of one month, although occasional samples show almost double this amount. At the end of two months the proportion of volatile acids, calculated as acetic acid, may exceed 0·5 per cent., and in some cases the presence of butyric acid now becomes marked at this period. The precise mechanism of the production of the volatile acids in sour milk is not very clear. Butyric acid is generally assumed to be derived from lactic acid, as shown in the equation:—

$$2CH_3.CH(OH).CO_2H = CH_3.(CH_2)_2.CO_2H + 2CO_2 + 2H_2;$$

while acetic acid may arise either from the oxidation of lactic acid, with the simultaneous evolution of carbon dioxide, or from the oxidation of ethyl alcohol, which is also an almost invariable constituent of sour milk. For each molecule of acetic acid produced there is formed in the one case a molecule each of carbon dioxide and of water, and in the other case only a molecule of water. In this latter instance, however, a molecule of carbon dioxide has already been lost in the production of the alcohol, so that the resultant loss is the same in either case. In the determination of the total solids of the sour milk, any acetic and butyric acids, together with any dissolved carbonic acid, are neutralized by the strontia used prior to evaporation, and are weighed with the non-fatty solids. The carbon dioxide which has escaped from the soured milk before neutralization, and the water and free hydrogen formed indirectly from the lactose, are, however, not weighed.

Ethyl alcohol is almost always formed in quantities, which in the above series of experiments ranged from 0·09 to 0·35 per cent. after eight weeks, during the fermentative changes which occur in milk under ordinary conditions. Although the conversion of lactose into alcohol is not brought about by ordinary yeast, yet certain of the *Schizomycetes* species effect the modification by the production first of dextrose and then of alcohol, with elimination of carbon dioxide. If, however, sufficient food for the organisms is present, galactose is also completely fermentable, so that the reaction in the case of lactose in milk may be expressed thus:

$$C_{12}H_{22}O_{11},H_2O = 4C_2H_5.OH + 4CO_2.$$

Again, the yeast ferments occasionally present in the market milk of large towns may also directly attack the dextrose produced in the course of the change from lactose to lactic acid. In any case, the production of one molecule of alcohol corresponds to the elimination of one molecule of carbon dioxide, so that the decrease in weight in the lactose, due to the production of the volatile alcohol and carbon dioxide, is $\frac{90}{46}$ times the weight of the alcohol produced.

Besides ethyl alcohol, there may also be found very small amounts of higher alcohols, glycerol, and succinic acid, in a manner analogous to the ordinary alcoholic fermentation. The amounts of these products can only be very small, and are negligible. Moreover, the higher alcohols produced would be taken account of in the analysis, being added to the ethyl alcohol, and any succinic acid would be neutralized and weighed with the solids of the milk.

The possible changes in the proteids of milk during fermentation are profound, but do not involve any considerable loss of weight. The ordinary "curdling" of milk in the early stages is simply a precipitation of the casein, due to the interaction of the lactic or acetic acid with the calcium proteid compound. Proteolytic changes, consequent on the development of enzyme-producing bacteria, result in the formation of a certain proportion of proteoses and peptones, and, later, of amino-compounds such as leucine, lysine and tyrosine; while small quantities of ammonia and amine bases may also be produced. The whole of these products, however, with the possible exceptions of traces of ammonia and amines, are weighed with the unchanged casein and other non-fatty solids in the analysis. On the one hand there may be a gain in weight due to the absorption of the elements of water in the hydrolysis of the casein molecule, and on the other hand there may be a loss due to the ultimate conversion of some of the casein into carbon dioxide, ammonia, and water.

Experiments in this direction show that, as regards the effect upon the weight of the solids, the net result of these changes is usually very small. The total loss of solid matter from all causes whatever ranges, as a rule, only from 0·2 to 0·5 per cent., and nearly the whole of this is accounted for by the production of alcohol and volatile acid from the lactose. Thus in the case of a series of watered and unwatered milks, the deficiency in solids, after allowing for that accounted for by the alcohol and volatile acid present, ranged from 0·00 to 0·07 per cent., after six weeks' keeping. During eight weeks, a series of experiments showed a production of ammonia and amines of 0·001 to 0·012 per cent., equal to a loss of, at most, 0·006 to 0·075 per cent. of proteids.

An investigation with about 20 gallons of milk was carried out by the author and his collaborators, with a view of obtaining further knowledge as to the nature and amount of the products formed during the souring of milk under the usual conditions. The milk was diluted with one-fourth of its volume of water, placed in stoppered bottles, and kept at a mean temperature of 18° C. during a period of ten to twelve weeks. At the end of that time the acid liquid was distilled, the distillate made alkaline and re-distilled. The distillation was again repeated, the liquid being rendered alternately acid and alkaline, and concentrated by means of a fractionating column. After further rectification and distillation, 76·4 grams of practically pure ethyl alcohol were recovered, and identified by the usual methods. The solution of the sodium salts of the volatile acids was examined, and found to contain a large quantity (about 390 grams) of normal butyric acid. Formic acid was absent, but acetic acid was identified (116 grams), and traces of propionic acid were probably also present. Less than 2 grams of an acid of higher molecular weight than butyric acid (possibly a nonoic acid) were obtained, and homologues of this acid were probably also present. A reserved portion of the fermented milk, freed from proteids and fat, gave on distillation with magnesia a small quantity of volatile bases which indubitably consisted very largely of ammonia. Hence, therefore, it may be considered proved that by far the greatest portion of the volatile products of the fermentation of milk, other than water and carbon dioxide, are ethyl alcohol, acetic and butyric acids, and a small quantity of ammonia. A small proportion of higher acids, possibly traces of propionic acid and of volatile organic bases, may also be produced. The amount of these by-products, in comparison with the main quantities, is so small that they are of no practical significance in this connection.

The great majority of samples dealt with by the referees under the Sale of Food and Drugs Acts are only fermented to a relatively small extent, being between three and six weeks old, and having undergone a loss of weight, in non-fatty solids, of from 0·2 to 0·5 per cent. Moreover, it should be borne in mind that in ordinary careful determinations of the non-fatty solids in the same sample of fresh milk by different persons the results may frequently differ by 0·1 to 0·2 per cent. Thus it may be assumed that a determination of this figure to within these limits is a satisfactory result, especially since an error of 0·1 per cent. only corresponds to about 1 per cent. in calculating the amount of added water in a sample.

The following methods of analysis are employed in the Government Laboratory· The "maceration" process is used, and the weight of the non-fatty solids and fat is independently ascertained in duplicate experiments, while, as a control, a direct determination of the total solids is made on a third portion of the milk.

The contents of the sample bottle are transferred to a suitable vessel and thoroughly mixed with a wire whisk. Portions of the sample, about 10 grams in each case, are weighed out into flat-bottomed platinum capsules, each of which has been tared along with a short glass rod having a flattened end. All the weighings and measurements are independently checked by two analysts. The weighed quantities are next neutralized with $\frac{N}{10}$ solution of strontia, using phenolphthalein as indicator, and the measure of strontia used noted. The milk is then evaporated over the water-bath until the residue, which towards the end should be dried at a very gentle heat and with constant stirring, attains the consistency of dry cheese. About 20 c.c. of dehydrated ether are next poured over the milk solids, which are then carefully triturated with the glass rod. The ethereal solution of the fat is passed through a filter, which has previously been dried and weighed in a weighing-bottle, and the maceration of the milk is continued with eight successive quantities of ether. At the conclusion of the process the non-fatty solids should be in a fine state of division, resembling the precipitated chalk of pharmacy. Before becoming quite dry, the solids are transferred as far as practicable to the weighing-bottle; the filter-paper, washed free from fat, is replaced in the bottle, and the whole, with the platinum capsule containing the small adherent quantity of solids, is dried at 100° C. for three hours and then weighed. The weight is again taken after drying for a further two hours, and a final confirmatory weighing after another hour. The last two weights should not differ by more than a mgm. Deducting 0·00428 gram for each 1 c.c. of decinormal strontia used in the neutralization, the result gives the amount of non-fatty solids actually present in the quantity of milk taken for the analysis. The ethereal solution of the milk-fat is received in small tared flasks, and after distillation of the ether the residue of dried fat is weighed.

From the judicial standpoint it is obviously desirable that the quantity of any constituent on which a legal charge may be based should be determined by direct weighing rather than by difference. The "maceration" process is the only one applicable to sour milks which leaves the residual non-fatty solids in a convenient form for accurate weighing. The trustworthiness of the determination of fat by this method has been completely established in the Government Laboratory. When conducted as described above, practically the whole of the fat is extracted, and is obtained in a form admitting of accurate determination. In the two following series of the results of comparison of the Bell maceration method with other processes, the first series was obtained by two analysts working independently, and are typical of forty analyses made to compare the "Werner-Schmid" and "maceration" methods; while the second series shows the results of H. D. Richmond's analyses of fresh milk by the "Adams' coil" and the "maceration" method respectively:

Percentages of Fat.

Werner-Schmid...	2·59	2·88	1·91	2·33	1·71	2·73	2·52	2·77	2·86
Maceration (Bell)	2·59	2·78	1·91	2·15	1·64	2·74	2·53	2·76	2·94
Werner-Schmid...	2·02	2·48	3·73	2·73	3·14	8·78	2·55	5·22	2·41
Maceration (Bell)	2·04	2·48	3·76	2·79	3·18	8·64	2·64	5·18	2·39
Adams	4·29	4·59	0·30	2·61	3·09	3·42	3·05	8·21	4·21
Maceration (Bell)	4·28	4·61	0·19	2·69	3·13	3·45	3·00	8·03	4·16

The following results (means of fifty-four analyses), carried out by three analysts working independently, were obtained on nine samples of fresh milk with a view of indicating the variations between the results given by the several methods (Minutes of Evidence, Departmental Committee on Milk and Cream Regulations, Blue Book· Cd. 484, p. 391):

Method.	Fat, Per Cent.	Method.	Fat, Per Cent.
Adams (dry ether)	3·76 ...	Maceration (Bell) ...	3·77
Adams (commercial ether)...	3·78 ...	Centrifugal ...	3·72
Werner-Schmid	3·88 ...	Centrifugal ...	3·72

Thus the figure yielded by the maceration method is exactly the average of all the others, and practically the same as that given by the Adams method.

For the determination of the alcohol, 50, 75, or 100 grams of the milk are distilled, and the distillate re-distilled after being neutralized with decinormal caustic soda solution, litmus-paper being used as the indicator. The specific gravity of the distillate, made up to the original or other convenient bulk, is determined in a 50-gram pycnometer, and the quantity of alcohol corresponding to this specific gravity is deduced from a table. The percentage by weight of alcohol, multiplied by $\frac{90}{46}$, gives the percentage of lactose which has disappeared in the production of the alcohol. The experimental error in the determination of these small quantities of alcohol is negligible, since the experience gained in the Government Laboratory shows that the differences obtained in successive tests of the same liquid would rarely or never exceed 0·00002 in terms of specific gravity, and usually would not be more than 0·00001. The higher of these figures would, in the case of milk, correspond to about 0·02 per cent. of non-fatty solids.

The proportion of volatile acid is ascertained by neutralizing 10 grams of the milk, contained in a platinum capsule, to the extent of one-half the total acidity (previously determined on another portion) with decinormal caustic soda, and a little phenolphthalein added. The mixture is then evaporated to dryness on a water-bath with frequent stirring, and after treatment with about 20 c.c. of boiling distilled water so as to break up and thoroughly detach the milk-solids from the capsule, a further addition of decinormal alkali is made, until the neutral point is reached. The difference between the original acidity of the milk and that of the evaporated portion is regarded as acetic acid. The production of 60 parts of acetic acid denotes a loss of 62 parts of the original lactose.

When it is desired to take account of any butyric acid that may be present, the volatile acids are separated by distillation from the quantity of milk that has been taken for determining the alcohol. A portion of the mixed aqueous solution of the acids is neutralized with baryta-water, evaporated, and dried until the weight is constant. From the percentage of barium contained in the mixed salts the proportions of the two acids are calculated. Each molecule of butyric acid (88 parts) denotes a loss of 92 parts of lactose. The loss of lactose due to the formation of acetic acid is calculated as before.

To estimate the small quantity of ammonia formed, 2 grams of the milk are made up to a volume of 100 c.c. with distilled water (ammonia-free), and filtered through a carefully-washed filter. In 10 c.c. of the clear filtrate, increased to 50 c.c. by the addition of ammonia-free distilled water, the ammonia is determined by Nessler's method, using a standard solution of ammonium chloride containing 0·01 mgm. of ammonia per 1 c.c.

The following results are typical of those given by the author in the original paper. The milks in question are those in which the butyric fermentation did not develop to any extent:

Sample No.	Weeks kept.	Loss of Non-fatty Solids.		Difference between Actual and Calculated Loss. Per Cent.
		I. Actual. Per Cent.	II. Calculated from Proportions of Alcohol, Acid, and Ammonia. Per Cent.	
		TABLE I.—UNWATERED WHOLE MILKS.		
1.	3	0·26	0·20	−0·06
,,	4	0·23	0·21	−0·02
,,	7	0·36	0·34	−0·02
,,	12	0·48	0·49	+0·01
5.	7½	0·26	0·20	−0·06
,,	11	0·49	0·49	0·00
,,	13	0·67	0·61	−0·06
6.	8	0·25	0·23	−0·02
7.	10½	0·27	0·35	+0·08
8.	11	0·36	0·32	−0·04
9.	13	0·87	0·76	−0·11
14.	2	0·19	0·16	−0·03
,,	4	0·23	0·18	−0·05
,,	6	0·20	0·17	−0·03
,,	8	0·30	0·20	−0·10
		TABLE II.—WATERED WHOLE MILKS.		
		1. *Approximately* 10 *Per Cent. of Water added.*		
1.	2	0·18	0·27	+0·09
,,	4	0·33	0·42	+0·09
2.	5½	0·36	0·33	−0·03
	5½	0·51	0·50	−0·01
		2. *Approximately* 25 *Per Cent. of Water added.*		
1.	2	0·13	0·13	0·00
	4	0·18	0·14	−0·04
	6	0·29	0 22	−0·07
	8	0·23	0·19	−0·04

TABLE III.—UNWATERED SEPARATED MILKS.

1.	4	0·41	0·22	−0·19
	5	0·32	0·13	−0·19
	9	0·31	0·26	−0·05
2.	4	0·29	0·28	−0·01
		0·31	0·20	−0·11

TABLE IV.—WATERED SEPARATED MILKS (*Approximately* 10 *Per Cent. Water*).

1.	3	0·32	0·24	−0·08
	55	1·47	1·50	+0·03
	55	1·54	1·56	+0·02
	57	1·47	1·50	+0·03

Thus the actual amount of original non-fatty solids may be very closely ascertained from the methods described, even although the keeping be very prolonged. The difference between the calculated and actual amounts of non-fatty solids is usually not greater than that obtained in duplicate determinations made by the same operator on fresh milk, and the proportion of added water may be correctly determined in fermented milk to within about 1 per cent. To simulate the worst conditions that might be found in practice, the 25 per cent. of water added in one series of experiments was a much-polluted sample from the old Fleet River.

The following figures show the results obtained on watered and unwatered milks in which butyric fermentation had set in:

TABLE V.

Sample No.	Weeks kept.	Butyric Acid. Per Cent.	Loss of Non-fatty Solids.		Difference. Per Cent.
			I. Actual. Per Cent.	II. Calculated. Per Cent.	
1	4	0·33	0·66	0·67	+ 0·01
,,	6	0·59	1·03	0·95	− 0·08
,,	8	0·55	0·86	0·90	+ 0·04
2	6	0·23	0·65	0·41	− 0·24
Approximately 25 per Cent. of Water added.					
3	4	0·17	0·34	0·22	− 0·12
,,	6	0·28	0·69	0·53	− 0·16
,,	8	0·66	1·39	1·10	− 0·29

The occurrence of butyric fermentation to any marked extent is infrequent amongst the samples received, but where it has occurred it will be seen from the above figures that the original non-fatty solids may be fairly accurately determined. Where the decomposition is considerable and much acid has been formed, it is sometimes difficult, on account of the separated casein, to get the sample into a uniform condition for analysis, in which case the examination is not carried out. So far as the butyric acid is concerned, however, this constituent may be readily determined on the principle already laid down, namely, by a determination of the proportion of barium in the mixed acids. A. R. T.

Note by Abstractor.—Standard strontia solution is preferred for the neutralization of the acidity of milk, because of the tendency of the precipitate to be less flocculent and to settle better. Thus the maceration method can be more rapidly carried out, and less precipitate finds its way on to the weighed filter-paper. The lower molecular weight of strontium also causes less error to be introduced, since any inaccuracy in the measurement of the solution has less influence on the amount of base to be deducted from the weight of the solids.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Saccharin in Wine. Ed. MacKay Chace. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1627.)—In order to eliminate the substance present in many wines which responds to the salicylic acid test for saccharin, the author uses the following method in testing for the latter substance: 50 c.c. of the wine are extracted as usual, the ether is evaporated off, and the extracted matter re-extracted with light petroleum. Whether or not the presence of salicylic acid is indicated, this extract is returned to the dish containing the residue from the extraction with light petroleum, the whole made up to about 10 c.c., and heated to boiling after adding 1 c.c. of sulphuric acid (1 : 3). If salicylic acid is present, an excess of a 5 per cent. solution of potassium permanganate is slowly added, and the boiling continued for one minute. To the hot solution a small piece of caustic soda is added, and the iron and manganese hydroxides are filtered off. The filtrate, which should be strongly alkaline, is transferred to a silver crucible lid, evaporated to dryness, and the residue heated to from 210° to 215° C. for twenty minutes. The melt is dissolved in a small quantity of water, acidulated with sulphuric acid (1 part in 3), and extracted with ether. The ether extract is tested for salicylic acid with a 0·5 per cent. solution of ferric alum.

The above method is capable of detecting 5 mgms. of saccharin per litre in the absence of salicylic acid. Salicylic acid is destroyed by it up to 200 mgms. per litre, and 10 mgms. of saccharin per litre can still be detected. A. G. L.

Determination of Butter-Fat and Cocoanut Oil in Margarine. A. Kirschner. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 65-70.)—A modification of a method first published by K. Jensen in the Danish Pharmaceutical Journal (*Farma. Tidende*, 1903, 385) is described. The method is a continuation of the Reichert-Meissl process, and is based on the precipitation of caprylic acid as insoluble silver caprylate, whilst silver butyrate is soluble. Five grammes of the sample are saponified and distilled in the usual way; 100 c.c. of the filtered distillate are then titrated with

$\frac{N}{10}$ barium hydroxide solution to obtain the Reichert-Meissl value. To the neutral solution 0·5 gramme of silver sulphate is added, and the mixture shaken from time to time for one hour. The solution is then passed through a filter, and 100 c.c. of the filtrate transferred to a distillation flask. After adding 35 c.c. of water, 10 c.c. of dilute sulphuric acid (1 : 40), and a few pieces of pumice-stone, the whole is distilled; 110 c.c. of distillate are collected, filtered, and 100 c.c. of the filtrate titrated with $\frac{N}{10}$ barium hydroxide solution. The number of c.c. of the latter required, calculated back to 5 grams of fat, is termed the "second titration" value. The results of analyses of mixtures of margarine with butter or cocoanut oil, or both, are given, from which results the author has worked out two formulæ for calculating the amounts of butter-fat and cocoanut oil present in any sample from the Reichert-Meissl and "second titration" values of the sample. The percentage of butter-fat $= 4{\cdot}319\,S - 0{\cdot}456\,R - 2{\cdot}15$, and the percentage of cocoanut oil $= 7{\cdot}42\,R - 8{\cdot}116\,S - 3{\cdot}57$; where $S =$ the "second titration" value and $R =$ the Reichert-Meissl value of the sample. W. P. S.

The Use of Talc as a Coating or Polishing Material for Pearl Barley, Rice, Millet, and Dried Peas. H. Matthes and F. Müller.

(*Zeit. öffentl. Chem.*, 1905, xi., 76-82.)—Of 25 samples of pearl barley examined by the authors, 16 were free from talc, 5 contained less than 0·2 per cent., and the remainder up to 0·63 per cent. Experiments carried out in the laboratory showed that by simply shaking the grains with talc it was quite possible to cause well over 2 per cent. of the latter to adhere to the barley. Samples containing talc may be detected by shaking the grains with water, allowing the mixture to settle, and examining the sediment under the microscope. The quantity of talc present may be ascertained by igniting 25 grams of the sample, extracting the ash with hydrochloric acid to remove magnesium compounds, etc., natural to the barley, and then analysing the residue. From 0·24 to 1·00 per cent. of talc was found in 15 samples of rice, whilst 8 samples were free from this substance. Samples of millet contained from 0·04 to 0·1 per cent. of talc, and peas from 0·082 to 0·188 per cent. W. P. S.

The Natural Alkalinity of Cocoa Ash. A. Froehner and H. Lührig.

(*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 257-263.)—The results of the analyses of cocoa beans of varying origin are given, from which it is seen that the water-soluble ash of 100 grams of dry cocoa powder requires from 10·0 to 30·0 c.c. of normal acid for its neutralization, and the insoluble ash from 30·0 to 42·7 c.c. By "cocoa powder" is meant ordinary commercial cocoa, containing less fat than cocoa beans. With regard to the question of added alkali in cocoa, it appears to be the general custom of manufacturers to treat "alkalized" cocoas with a quantity of alkali equivalent to less than 3 per cent. of potassium carbonate. The authors considered that the alkalinity of a cocoa which requires less than 74 c.c. of normal acid to neutralize the soluble ash and 114 c.c. for the insoluble ash from 100 grams of cocoa does not exceed this limit. It is important to determine the alkalinity of the insoluble ash, as some makers use magnesium carbonate instead of, or in conjunction with, potassium and sodium carbonates. W. P. S.

Sugar as a Natural Constituent of Mace. W. Ludwig and H. Haupt. (*Zeit. Untersuch. Nahr. Genussmittel,* 1905, ix., 200-204.) — Having recently had occasion to determine the quantity of added sugar in samples of adulterated mace, the authors first proceeded to ascertain whether the arillus or "mace" of the *Myristicaceæ* naturally contained sugar. The maces examined were of various kinds—Banda, Menado, Papua, and Bombay—and were thoroughly extracted with petroleum spirit before dissolving out the sugary substances with water. The results obtained are given in the following table, the polariscopic readings being made on a solution containing 26·048 grams of the original substance per 100 c.c.:

Kind of Mace.	Polarization in a 200-millimetre Tube.	Sugar (as Dextrose) by Fehling's Method.
Banda	+8·0	2·80 per cent.
Banda	+8·4	4·28 ,,
Menado	+6·8	2·19 ,,
Papua	+6·4	1·65 ,,
Bombay	+0·8	2·34 ,,

The aqueous sugar solution underwent scarcely any change when inverted with hydrochloric acid at a temperature of 100° C.

Two adulterated samples gave polariscopic readings of +15·2° and +36·4° respectively. The first, as the result of further investigation, was found to contain 9·5 per cent. of lactose and the second a considerable quantity of dextrin. The copper reductions of this latter sample, calculated as dextrose, were: Direct, 4·26 per cent.; inverted at 60° C., 4·61 per cent.; and inverted at 100° C., 17·54 per cent.

W. P. S.

The Adulteration of Saffron. A. Nestler. (*Zeit. Untersuch. Nahr. Genussmittel,* 1905, ix., 337-344.)—The peculiar crystals noticed in some samples of saffron, and previously mentioned by the author (Analyst, 1904, 94), have now been found to consist of sugar, probably added as an adulterant. Two samples are also mentioned: one being adulterated with 12·9 per cent. of barium sulphate and the other with 33·4 per cent. of a mixture of potassium nitrate and borax. W. P. S.

The Determination of Morphine in Opium. Arthur and Albert Petit. (*Journ. Pharm. Chim.,* 1905, xxi., 107-111.)—The following method is that adopted as the official one in the new edition of the French Codex: 15 grams of an average sample of the opium are triturated in a mortar with 6 grams of slaked lime, and then mixed with 150 c.c. of water and allowed to stand for two hours with occasional agitation. After this the mixture is transferred to a filter, and 106 c.c. of the filtrate (=10 grams of opium) collected. This is shaken with 30 c.c. of ether (specific gravity 0·725) so as to saturate the aqueous layer. Two grams of ammonium chloride (free from carbonate) are next dissolved in the liquid, which is shaken until a distinct precipitate appears, and then left for twenty-four hours in a covered beaker. The ethereal layer is then decanted on to two small counterpoised filters, and the aqueous portion again shaken with 30 c.c. of ether, which is decanted in turn. Finally the aqueous layer is poured on to the filters, so that the whole of the precipitate is collected. The filters are washed with 25 to 30 c.c. of water previously saturated with morphine, and dried for two hours at 100° C. They are then washed with 20 c.c. of chloroform (deprived of alcohol by shaking with water) until the filtrate is colourless, after which the residue of morphine is again dried and weighed. The morphine thus purified ought to be completely soluble in $\frac{N}{10}$ sodium hydroxide solution. Opium analysed by this method should yield not less than 10 per cent. of morphine. The authors confirm the statement of Dott and Hesse that morphine retains one molecule of water of crystallization even after prolonged drying at 100° C. C. A. M.

Determination of the Purity of Iodine. Hennecke. (*Pharm. Zeit.*, xlix., 957; through *Pharm. Journ.*, 1904, lxxiii., 958.)—The German and British Pharmacopœias direct that the purity of iodine be determined by dissolving it in potassium iodide solution and titrating with standard sodium thiosulphate solution in the usual manner. Weinland has recently pointed out that any iodine chloride present in commercial iodine will react with the potassium iodide as follows: $ICl + KI = KCl + I_2$, the liberated iodine being also titrated. The author modifies the official method by dissolving the iodine in dry chloroform, and titrating the liquid by means of sodium thiosulphate, the end-reaction being quite definite without the aid of starch solution. In the absence of water and potassium iodide, the iodine chloride is unacted on and takes no part in the reaction. A sample of commercial iodine which showed 100·9 per cent. of iodine by the pharmacopœial process gave 99·0 per cent. by the chloroform method. A. R. T.

Cod-liver Oil Standards. E. J. Parry. (*Chemist and Druggist*, 1905, lxvi., 491, 492.)—The tabulated results of the analyses of forty samples of cod-liver oil are given. The samples include the best brands known to the market, and no samples of doubtful origin have been included. The specific gravities (60° F.) of the samples were all within the limits 0·924 and 0·931. Most of the samples contained less than 1 per cent. of free acid, those containing more than 1 per cent. usually having a very rancid odour. Using Hübl's solution in excess for eighteen hours, the iodine values found varied from 159 to 169. The Reichert values were generally below 0·7, although some undoubtedly pure oils gave values as high as 1·0 (for 2·5 grams). All the oils of good quality contained less than 1·5 per cent. of unsaponifiable matter. A few samples exceeded this figure, but they were rancid, and could not be accepted as of medicinal quality. The author considers that the high percentages of unsaponifiable matter found by Mann (ANALYST, 1904, 93) are incorrect. W. P. S.

The Occurrence of Arsenic in Hydrogen Peroxide. L. Grimbert. (*Journ. Pharm. Chim.*, 1905, xxi., 385, 386.)—According to the author, arsenic is frequently present in commercial solutions of hydrogen peroxide. One sample recently examined gave a red precipitate on the addition of silver nitrate. When heated on the water-bath with a solution of calcium hypophosphite in hydrochloric acid it rapidly turned brown, and yielded a deposit of arsenic, the amount of which corresponded to 0·202 gram per litre. The residue left on evaporation of the hydrogen peroxide was 2·85 grams per litre, of which 2·10 grams consisted of sodium chloride, and the remainder of arsenates and sulphates. C. A. M.

ORGANIC ANALYSIS.

Potassium Oxalate as a Lead Precipitant in Sugar Analysis. Harris E. Sawyer. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1631.)—The author has found that potassium oxalate precipitates lead completely and easily from sugar solutions, without altering the reducing power towards Fehling's solution in any way. He uses 6 c.c. of a $\frac{2N}{1}$ normal potassium oxalate solution for every 50 c.c. of a solution obtained by dissolving 26 grams of sugar in 500 c.c. of water, adding 8 c.c. of a solution of basic lead acetate, and filtering after a few minutes' shaking. Sodium oxalate may also be used, but the more soluble potassium salt is to be preferred. A. G. L.

The Determination of Phenol. S. J. Lloyd. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 16.)—As a result of a study of the sources of error in the determination of phenol by means of bromine, the author recommends the following procedure: The phenol solution is placed in a glass-stoppered flask, and to it is added a volume of hydrochloric acid (specific gravity 1·2) equal to about one-third or one-fourth of the combined volumes of the phenol solution and the hypobromite solution subsequently added. (This hypobromite solution is prepared by dissolving 9 c.c. of bromine in 2 litres of a solution containing 28 grams of potassium hydrate; it is standardized by adding acid and potassium iodide and titrating with $\frac{N}{50}$ thiosulphate solution.) The hypobromite solution is added to the acidified phenol solution until the solution becomes permanently yellow; an additional 10 or 20 per cent. of the hypobromite is then added, and the whole well shaken. An excess of potassium iodide is added, followed by about 10 c.c. of water for every 1 c.c. of acid present; after adding 10 c.c. of chloroform, the iodine liberated is titrated with thiosulphate. The object of diluting with water is to prevent the acid from acting on the iodide or thiosulphate; the presence of chloroform ensures more uniform results. Sulphuric acid may be substituted for the hydrochloric acid without impairing the accuracy of the determination. From the test analyses it appears that the method yields results accurate to about 0·2 per cent. A. G. L.

Tribromphenolbromide: its Detection, Estimation, Rate of Formation, and Reaction with Hydriodic Acid. S. J. Lloyd. (*Journ. Amer. Chem. Soc.*, xxvii., 7.)—The author finds that tribromphenolbromide ($C_6H_2Br_3OBr$) is not the primary product of the action of bromine on phenol, but is formed by the gradual action of bromine water on the first formed tribromphenol. As the rate of this reaction is decreased by adding acid or potassium bromide, and increased by adding water or bromine, or by raising the temperature, it is probably due to the presence of hypobromous acid in the bromine water. Aniline and benzidine in chloroform solution form convenient reagents for the detection of tribromphenolbromide. Aniline gives a deep-red colour, turning muddy; benzidine, an intense green, or purple if concentrated; with tribromphenol, tetrabromphenol, and hexabromphenoquinone, neither aniline nor benzidine gives a colour; bromine water bleaches aniline and gives a seal-brown colour with benzidine. The following colour reactions of tribromphenolbromide are less distinctive: Ammonia gives a brown colour; dimethylaniline, slight darkening; paratoluidine, clear red; diphenylamine, light red; α-naphthylamine, dark blue, turning purple; β-naphthylamine, light-rose pink, which appears very slowly; aminoazobenzene, brown-red; pyridine, light yellow; azoxybenzene, *m*-nitraniline, and acetanilide produce no change. The presence of a little sodium thiosulphate does not interfere with the reactions.

Tribromphenolbromide is not quantitatively reduced to tribromphenol by hydriodic acid unless the solution is exceedingly dilute. Powdered zinc and sulphuric acid, on the other hand, reduce it quantitatively. The reaction is conveniently carried out by placing about 0·5 gram of the substance in a 150 c.c. flask, and heating on a water-bath with 20 c.c. of 96 per cent. alcohol, 5 c.c. $\frac{N}{4}$ sulphuric acid, and 0·25 gram powdered zinc, until the solution has lost its characteristic colour. Excess of calcium carbonate is then added; the alcohol is distilled off on a water-bath, and the residue extracted three times with boiling water, using 25 c.c., 25 c.c., and 10 c.c., respectively. In the filtrate the bromide is titrated with silver nitrate and potassium chromate. Three tests made in this way gave 99·92, 100·61, and 99·07 per cent. of tribromphenolbromide recovered. A. G. L.

A New Colour Reagent of the Polyphenols, their Isomers, and Higher Organic Compounds. E. P. Alvarez. (*Chem. News*, 1905, xci., 125.)—In a small porcelain basin are placed 0·2 gram of perfectly dry sodium dioxide, immediately afterwards 0·04 gram of the polyphenol to be tested, and then 5 c.c. of pure anhydrous ethyl alcohol. The contents of the basin are gently agitated for about five minutes, after which 15 c.c. of cold water are added. If the water be added at once the mixture may ignite. Colorations are obtained before and after adding the water, and in some cases an "edge coloration" is obtained by breathing on the edge of the liquid before diluting. The following colorations have been observed:

	Sodium Peroxide + Alcohol.	After breathing on Edge of Mixture.	After adding the Water.
Pyrocatechol.	Transitory pale pink, becoming green, then brown.	Blue-green.	Permanent red-brown.
Resorcinol.	Very pale yellow, becoming green.	—	Permanent deep green.
Hydroquinone.	Intense reddish-yellow.	Transitory blue.	Persistent orange.
Pyrogallol.	Dull red.	—	Intense red.
Oxy-hydroquinone.	Reddish-violet, changing to dark brown.	—	Yellowish.
Phloroglucinol.	Blue-violet.	—	Intense blue-violet, which slowly fades.
Orcinol.	Intense pink.	—	Rose red.
Homopyrocatechol.	Blue-violet, changing at once to red.	Greenish-yellow.	Reddish-brown.
Thymohydroquinone.	Intense orange.	Deep yellow.	Wine red, slowly fading.

W. P. S.

The Rotatory Power of Oil of Turpentine. L. Raby. (*Ann. de Chim. anal.*, 1905, x., 146-147.)—Riban has shown that on distilling neutral anhydrous oil of turpentine the sum of the rotatory powers of the different fractions is exactly that of the original oil. This is not the case, however, if the turpentine be acid or contain moisture. Thus, a commercial sample examined by the author yielded four fractions and a residue, each of which had a rotatory power lower than that of the original oil. After four months the rotatory powers of the four distillates had risen considerably, whilst that of the residue had not materially altered. By diluting one of the freshly-distilled fractions with its own volume of inactive olive oil the rotatory power immediately rose to the value that it only slowly attained in the case of the pure oil. The original oil had the specific gravity of 0·873 at 12° C., and its rotatory power (α_D) was − 34·19°. It was moist and acid, and did not show any trace of resinification. The results obtained on distillation were as follows:

	Boiling-point, °C.	Specific Gravity at 12°C.	Rotatory Power (α_D).	
			Immediately after Distillation.	Four Months after Distillation.
First Fraction ..	153 -154·5	0·8693	− 28·93°	− 37·66°
ditto + olive oil	—	—	− 37·65°	− 37·65°
Second Fraction ..	154·5-155	0·8697	− 28·63°	− 35·85°
Third ,, ..	155 -156	0·8702	− 28·03°	− 34·90°
Fourth ,, ..	156 -158	0·8709	− 26·98°	− 34·62°
Fifth (residue) ..	above 158	0·8951	− 23·29°	− 23·16°

C. A. M.

A New Method of Determining the Viscosity of Light-coloured Mineral Oils. Rudolf Nettel. (*Chem. Zeit.*, 1905, xxix., 385.)—The method depends on noting the time it takes a drop of water to fall through a definite height of the oil whose viscosity is to be determined. The time required is a function of the viscosity and the specific gravity of the oil; if the latter be known, then by comparison with oils of known specific gravity and viscosity, the unknown viscosity may be calculated. In carrying out the method, the time taken for a drop of water to fall through a definite height in two oils, with known viscosities, which should be as widely apart as possible, is first noted. Let F_A and F_a represent the quotient

$$\frac{\text{Time of falling}}{\text{Viscosity according to Engler}}$$

of the two oils, A and a, respectively. Then F_x—*i.e.*, the corresponding value for an oil of unknown viscosity—is given by the equation:

$$F_x = F_A - \left[\frac{F_A - F_a}{\text{Sp. gr. A} - \text{Sp. gr. a}} \times (\text{Sp. gr. A} - \text{Sp. gr. } x)\right].$$

And if the time of falling through the same height in the oil x be determined, the viscosity in Engler degrees is obtained by dividing this time by F_x.

The determination of the time of falling may be conveniently made in a burette of about 12 millimetres diameter; the bore must be very even throughout the tube.

The height may be 400 millimetres, or, for very thick oils, one quarter of this. The standard oils and the height are chosen so that the fraction

$$\left(\frac{\text{Fa} - \text{Fa}}{\text{Sp. gr. A} - \text{Sp. gr. a}}\right)$$

equals 100, which simplifies the calculation. The "Vereinigten Fabriken für Laboratoriumsbedarf" in Berlin make an apparatus in which a number of tubes are mounted in a water-jacket, the use of which appears to be essential, since an alteration of 1° in the temperature causes a large difference in the time of falling. In seven test results quoted the maximum difference between the viscosity as directly determined and as calculated according to this method is 0·2. Of course, care must be taken that the drops of water used are always of the same size. A. G. L.

Composition of Grape-seed Oil. F. Ulzer and K. Zumpfe. (*Oester. Chem. Zeit.*, 1905, viii., 121-123.)—The following chemical and physical constants were yielded by a sample of this oil:

Specific gravity at 15° C.	0·9215
Saponification value	190·0
Iodine value	142·8
Maumené value	81° to 83° C.
Refractometer (Zeiss) reading at 50° C. ...	54·5
Refractive index	1·4623
Acetyl value of the fatty acids	43·7

The authors also fractionated the solid fatty acids—separated from the fluid fatty acids by the lead method—and from the further examination of these came to the conclusion that the oil consists chiefly of the glyceride of linolic acid, about 10 per cent. of solid glycerides and smaller quantities of the glycerides of oleic, ricinoleic, and linolenic being also present. Erucic acid could not be detected, and the oil certainly contained only the smallest traces, if any, of this acid. W. P. S.

On the Alleged Natural Occurrence of Heptadecylic Acid. D. Holde. (*Berichte*, 1905, xxxviii., 1247-1248.)—*Olive Oil.*—The author previously came to the conclusion that a mixed glyceride in olive oil contained a heptadecylic acid, $C_{17}H_{34}O_2$ (ANALYST, xxvi., 324). He has since examined a larger amount of this substance, fractionally precipitating it with magnesium acetate, and after 15 fractionations succeeded in isolating palmitic acid and other fatty acids, some of which had a molecular equivalent above 290. An artificial mixture of arachidic acid (0·3 gram), stearic acid (0·7 gram), and palmitic acid (1 gram), behaved similarly to the acids from the mixed glyceride, yielding when fractionated four successive fractions, whose melting-points and molecular equivalents agreed closely with those of the supposed heptadecylic acid. Hence it is not safe to conclude that a substance is a chemical individual even when four or five successive fractions are nearly constant in melting-point and molecular equivalent. The solid fatty acids of the olive oil itself were also fractionated, and after ten crystallizations an acid of molecular equivalent 368·7 and melting-point 72° to 72·8° C. was separated (lignoceric acid melts at 80·5° C., and has a molecular equivalent of 368).

Kreis and Hafner's Heptadecylic Acid.—The actual preparation of Kreis and

Hafner (Analyst, xxviii., 359), which melted at 55·5° to 56·6° C., was examined in the same way, and eventually, after a large number of fractionations, it yielded stearic acid melting at 68° to 69° C., and having a molecular equivalent of 280·1.

Gérard's "Daturic" Acid.—The acid isolated by Gérard from datura oil (*Comptes Rend.*, 1890, iii., 305), and the existence of which was confirmed by the author (*Mitt aus dem königl. techn. Versuchsamt*, 1902, xx., 66), has also been fractionated in large quantities. When fractionally distilled *in vacuo*, and the products of the distillation crystallized repeatedly from alcohol, this supposed acid now yielded a fatty acid with a molecular equivalent of 329 and melting-points of 57°–58·5° C. to 63° to 64° C.

Nördlinger's Heptadecylic Acid.—The substance prepared by Nördlinger (*Zeit. angew. Chem.*, 1892, 110) from palm oil was examined in a similar way, large quantities being used for the fractionation. Several successive fractions showed an apparently constant melting-point of 56° C., and the molecular equivalent of heptadecylic acid, but eventually, after repeated fractionation, an acid melting at 68° to 68·5° C., and with a molecular equivalent of 288, was isolated. The apparent constancy of the earlier fractions was attributed by the author to there being present an intimate admixture of palmitic acid with acids of very high molecular weight.

C. A. M.

A Note on the Detection of Archil, Cudbear, and Other Lichen Colours.

L. M. Tolman. (*Journ. Amer. Chem. Soc.*, xxvii., 25.)—In testing foods and medicines for added colours by dyeing a piece of wool in an acid-bath, extracting the colour with ammonia, acidifying this solution, and dyeing a second piece of wool, the lichen colours, as well as coal-tar colours, will be collected on the second piece of wool. They may be differentiated, however, by separating them in the first place from the natural colouring matters of any fruits or wines which may be present by extracting the ammoniacal solution with amyl alcohol, the alcoholic extract being evaporated and the purified colour tested. An aqueous solution of the lichen colours is readily reduced by tin and hydrochloric acid, and reoxidized by ferric chloride, being thus distinguished from the azo-dyes and magenta, which are used as substitutes. For further identification, if thought necessary, the colour can be treated according to Allen (Vol. III., Part I., pp. 525 to 541). Sulphonated orceine, as well as the other forms in which lichen colours are used, gives the above reactions. A. G. L.

The Nitrosites of Caoutchouc and their Application in the Analysis of Crude Caoutchouc and of Caoutchouc Goods.

Paul Alexander. (*Zeits. angew. Chem.*, 1905, xviii., 164.)—The author has analysed the so-called dinitro-compound produced according to Weber's method (*Ber.*, xxxvi., 3103) from twenty-eight different samples of caoutchouc, and finds its mean composition to be, for South American samples, C 45·98, H 5·45, N 11·63 per cent.; and for African samples, C 44·33, H 5·77, N 12·06 per cent.; whilst Weber's formula $C_{10}H_{10}N_2O_4$ requires C 52·63, H 7·02, N 12·28 per cent. The author believes the dinitro-compound to consist of a mixture of the nitrosites of Harries with oxidized derivatives of caoutchouc. A. G. L

On the Analysis of Caoutchouc and Gutta-percha. W. Esch and A. Chwolles. (*Chem. Zeit.*, 1904, xxviii., 1195.)—From a consideration of the methods employed and the values obtained by C. Harries (*Berichte*, 1903, xxxvi., 1937), the authors believe his "nitrosite c." method to be unreliable, if not worthless, especially as regards his failure to take into account the resinous and slimy substances always present in Para caoutchouc. They also believe that at present a chemical analysis is of little value in judging of the properties of a sample of rubber.

A. G. L.

Detection of Adulteration in Natural Asphalt. B. Malenkovic. (*Oester. Chem. Zeit.*, 1905, viii., 123-126.)—The following qualitative tests are described for distinguishing between asphalt and such substances as petroleum pitch, coal-tar pitch, and wood pitch. By determining the bromine value, the author also attempted to approximately determine the amounts of these substances in mixtures of the same. In all the tests the asphalt was extracted with carbon disulphide, and the experiment performed on the extracted bitumen after drying the latter for thirty minutes at 105° C.

Solubility in Sodium Hydroxide Solution.—One gram of each substance was boiled for fifteen minutes with 100 c.c. of 10 per cent. sodium hydroxide solution, and the colour of the solution observed. Asphalt (Syrian, Dalmatian, and Trinidad) gave a nut-brown coloration; petroleum pitch, none; coal-tar pitch, light yellow; and wood pitch, reddish-brown.

Solubility in Formaldehyde.—When boiled for fifteen minutes with a 40 per cent. formaldehyde solution asphalt yielded no soluble coloured matter, whilst all the surrogates gave coloured solutions.

Solubility in Alcohol.—One gram of the substance was dissolved in 10 c.c. of carbon disulphide, placed in a 100 c.c. cylinder, and treated with 90 c.c. of petroleum spirit. After filtration, a portion of the filtrate was mixed with 85 per cent. alcohol and gently shaken, but not sufficiently so to form an emulsion. With asphalt the alcoholic layer remained colourless, whilst in every other case it was coloured yellow From 2 to 5 per cent. of adulteration could readily be detected by means of this test.

Bromine Value.—About 0·5 gram of the substance was placed in a small flask two-thirds filled with carbon tetrachloride, and treated with 25 c.c. of anhydrous bromine. After heating under a reflux condenser for about twenty-five hours, the contents of the flask were poured into a large porcelain basin and evaporated. The residue was dissolved in carbon tetrachloride, transferred to a tared flask, again evaporated, dried for thirty minutes at 105° C., and weighed. The following quantities of bromine were found to be absorbed (by addition and substitution): Trinidad asphalt, 93 per cent.; Dalmatian asphalt, 89 per cent.; petroleum pitch, from 62 to 72·5 per cent.; coal-tar pitch, from 173 to 191 per cent. W. P. S.

The Determination of Organic Carbon in Soils. J. H. Pettit and T. O. Schaub. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1640.)—The author has obtained exceedingly good and rapid results by the use of Parr's method (*Journ. Amer. Chem. Soc.*, xxvi., 294) of combustion with sodium peroxide, with subsequent determination of the carbonate formed. In four determinations on pure sugar, from 99·46 to 99·89 per cent. of the carbon present was found. In dealing with soils, the author used magnesium to start the reaction, a mixture of 1 gram of finely-ground magnesium powder with 2 grams of soil and 10 grams of sodium peroxide being used. The objection to the use of sulphur is that sulphur dioxide was always present in the gases evolved by treating the residue in the bomb with acid, even when potassium nitrate was added to the charge. As the magnesium is not always completely oxidized, and any residual metallic magnesium liberates hydrogen on treatment with acid, the carbon dioxide, after measuring, is absorbed by caustic potash and any residual hydrogen measured and taken into account. Results obtained in this way were practically identical with those given by combustion with copper oxide.

A. G. L.

INORGANIC ANALYSIS.

The Analysis of Tin Samples. Ernst Victor. (*Chem. Zeit.*, 1904, xxix., 179.)—A description of a very complete method for the evaluation of commercial tin samples: 10 to 20 grams of the sample are dissolved in an Erlenmeyer flask in 100 c.c. of hydrochloric acid (specific gravity 1·124). After cooling, small quantities of potassium chlorate are added until the metals which had remained undissolved (Cu, Sb, As) are entirely in solution, and the solution boiled until all chlorine is eliminated. Ammonium tartrate, prepared by dissolving 30 grams of pure tartaric acid in a little warm water, and, after cooling, adding excess of ammonia, is added to the hydrochloric solution, and this then rendered just alkaline with ammonia. Cu, Pb, Sb, and Fe are then precipitated by the addition of small quantities of H_2S water, boiled between each addition, filtered off and washed with H_2S water. The lead is then determined electrolytically in the following manner: The mixed sulphides are dissolved in warm nitric acid (specific gravity 1·2), heated until the precipitated sulphur just turns yellow, filtered into a platinum dish, and the lead separated electrolytically as the peroxide, using a current of 0·2 to 2 ampères.

For the determination of copper, a separate portion of the sample is treated in a similar manner, rendering the nitric acid solution of the sulphides strongly ammoniacal. The precipitated hydroxides are filtered off, and the filtrate, acidified with nitric acid, is electrolysed in the usual manner. Iron is separated as the hydroxide from the solution from which the lead was electrolytically separated.

For the determination of antimony, a further portion is dissolved and oxidized as above. The chlorine is boiled off, the solution considerably diluted, and a few clean iron nails and a very little reduced iron are added. The flask, closed with a Bunsen valve, is heated on a sand-bath until the Sb, Cu, and As are separated in metallic form. The flask, avoiding the ingress of air, is cooled and the solution filtered through a paper on which a little reduced iron has been placed, the filter washed very thoroughly with water acid with HCl, and the deposit washed back into the flask. To obtain complete solution of this deposit, a little chlorate is placed on the filter, and this then washed with hot hydrochloric acid. The flask is gently warmed until solution is complete, when small fragments of solid sodium hydrate are added until a distinct excess is present. The solution is filtered through asbestos, 50 c.c. of saturated sodium sulphide solution added, and, after boiling, it is again filtered.

Antimony is separated from this filtrate by electrolysis, using a current of 0·2 to 1 ampère.

The iron solution is prepared by dissolving 27·5 grams of ferric chloride with 25 c.c. of concentrated hydrochloric acid and making up to 1 litre; each c.c. of this solution corresponds to 0·01 gram of Sn. As indicator, starch and a special iodine solution are used; this is prepared by adding 10 grams of potassium iodide, dissolved in 10 c.c. water, to 10 grams of hydriodic acid (specific gravity 1·5) containing 3·3 grams of iodide of copper. This solution should be kept in the dark for some days before use.

For the titration, 5 grams of the sample are dissolved in 500 c.c. of hydrochloric acid, adding chlorate to complete the oxidation. Fifty c.c. are filtered through asbestos into a flask, and, by means of a doubly perforated stopper, CO_2 is passed into the air-space in the flask, and some coarse aluminium powder is added. After complete solution of the aluminium and precipitation of the tin, further 50 c.c. of hydrochloric acid are added, and the whole heated until solution is complete. The solution is cooled in a current of CO_2 and titrated with the iron solution, using 10 drops of the iodine indicator and a little starch solution.

This method is correct to 0·2 per cent. with samples containing less than 97 per cent. of Sn.

NOTE.—Th. Goldschmidt, in a letter to the *Chemiker Zeitung* (1905, xxix., 276), draws attention to the fact that the above titration method has been employed in his laboratory (Staatshütten Laboratory, Essen a.d. Ruhr) for the past fifteen years, and that the author of this paper, E. Victor, had worked in this laboratory in 1900-1901. H. A. T.

A Characteristic Reaction of Cobalt. E. Pozzi-Escot. (*Ann. de Chim. anal.*, 1905, x., 147.)—Solutions of cobalt salts yield insoluble reddish-brown precipitates on treatment with mono- substituted thiohydantoic acids or their derivatives. Thus, on adding a few drops of an alcoholic solution of phenyl- or β-naphthyl-thiohydantoic acid, and then 1 drop of ammonium hydroxide, to a very dilute solution of a cobalt salt, there is an immediate scarlet coloration. Salts of nickel, under the same conditions, give an ochre yellow coloration or a grey precipitate. This coloration completely masks that of the cobalt if also present, but on adding an excess of ammonium hydroxide the nickel salt is dissolved instantaneously, yielding a colourless liquid, whereas the cobalt salt is much less soluble, and sufficient will be left undissolved to show the red coloration. C. A. M.

The Use of a Rotating Anode in the Electrolytic Estimation of Zinc. Leslie H. Ingham. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1269.)—The author obtained good results in the electrolytic deposition of zinc from various solutions, using a platinum spiral revolving at the rate of 230 or 560 revolutions per minute as anode. The cathode consisted of a silvered dish, from which the zinc was removed at the end of each experiment by dilute sulphuric acid (1 : 50). It was found that, using an electrolyte containing sodium acetate, 0·5 gram zinc would be completely deposited with the higher speed in fifteen minutes, using a current of 4 ampères (for about 100 square centimetres) and 11 volts. With sodium hydroxide solutions

0·25 gram zinc could be deposited in fifteen minutes, using 5 ampères and 6 volts. From sodium formate solutions slightly acid with formic acid, 0·25 gram zinc were deposited in twenty minutes, using 5 ampères and 8 volts. Potassium cyanide solutions gave unsatisfactory results.

Both the sodium acetate and formate methods were found to be especially applicable to the determination of zinc in zinc blende, the time required to complete the analysis on 0·5 gram of ore being only about two and a half hours. In each case the iron was removed by two precipitations as basic acetate or formate. The best conditions for the composition of the final mixed filtrate, which was used as electrolyte, were found to be: (*a*) Dilution, 125 c.c.; $NaC_2H_3O_2$, 3·5 to 4·5 grams; current, 5 ampères and 13 volts; time, thirty minutes. (*b*) Dilution, 125 c.c.; Na_2CO_3, 5·5 grams; formic acid (specific gravity 1·22), 4·8 c.c.; current, 5 ampères and 9 volts; time, twenty-five minutes. (The Na_2CO_3 in (*b*) represents the salt used to neutralize the acid in the solution before the basic formate precipitation.) Working on a zinc blende containing 65·70 per cent. of zinc, the results of ten experiments by both methods varied between 65·62 and 65·96 per cent.

It was not found possible to use the sodium hydroxide method for the determination of zinc in the blende, as the separated ferric hydroxide always contained zinc; but good results were obtained by substituting ammonia for the sodium hydroxide, the iron being separated by two precipitations, and the mixed filtrates electrolysed under the following conditions: Dilution, 125 c.c.; hydrochloric acid (specific gravity 1·21), 10 c.c.; ammonia (specific gravity 0·95), 2 c.c. in excess; NH_4Cl, 0·5 gram; current, 5 ampères and 6 volts; time, twenty minutes. The results varied between 65·63 and 65·75 per cent. The anode was not attacked during these experiments. A. G. L.

The Preparation of Pure Earths of the Cerium Group by Means of their Double Alkali Carbonates. R. J. Meyer. (*Zeits. anorg. Chem.*, 1904, xli., 97.)—The preparation of double carbonates of cerium, lanthanum, praseodymium, and neodymium with the alkalis is described. These are generally of the types $K_2La_2(CO_3)_4.12H_2O$; $(NH_4)_2La_2(CO_3)_4.4H_2O$; and $Na_6La_4(CO_3)_9.22H_2O$; but ammonium cerium carbonate has $6H_2O$, and all the sodium compounds are so easily decomposed that their exact composition, especially as regards the amount of water they contain, is doubtful. All the carbonates are obtained by adding a solution of a salt of the rare earth to a concentrated solution of an alkali carbonate; the sodium and ammonium compounds are practically insoluble, whilst the potassium compounds are easily soluble in strong potassium carbonate solution. On diluting this solution with water, the double carbonates are precipitated in the following order: Lanthanum, praseodymium, cerium, neodymium. This behaviour affords a ready means of purifying the rare earths. Job's method (*Compt. rend.*, 1898, cxxvi., 246) is recommended as a delicate test for cerium, 0·5 per cent. of this earth in mixtures with other rare earths being still recognisable. The test consists in dissolving the earths in potassium carbonate and adding a little hydrogen peroxide, a yellow colour indicating cerium.

The praseodymium peroxide obtained by ignition of the oxalate corresponded most nearly with the formula Pr_6O_{11}. The oxide PrO_2 was obtained, following

Brauner's directions, by heating the nitrate with potassium nitrate to 400° to 450° C. It could not be obtained by heating Pr_6O_{11} with CeO_2. Baskerville and Turrentine's citric acid method (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 46) for the purification of praseodymium was found to be impracticable. A. G. L.

A New Method of Detecting and Determining Ammonia. A. Trillat and Turchet. (*Bull. Soc. Chim.*, 1905, xxxiii., 304-310.)—Nessler's reagent gives erroneous results in the presence of hydrogen sulphide, carbonic acid, lime, and certain albuminoid substances, owing to appreciable quantities of ammonia being thereby masked. The author's new method is claimed to obviate these sources of error, while being as sensitive and rapid as Nessler's process. It is based upon the formation of nitrogen iodide, which gives an intense black coloration to water, easily perceptible in a solution containing 1 part of ammonia in 500,000. Nitrogen iodide is not produced in very dilute solutions by the direct action of iodine on ammonia, but is instantly formed by the action of iodine chloride on ammonia, and the compound is stable in the presence of an alkali:

$$3ClI + NH_3 + 3NaOH = 3NaCl + NI_3 + 3H_2O.$$

Thus, if a water containing ammonia be treated with a solution of potassium iodide, and a few drops of a dilute solution of an alkali hypochlorite, the iodide is decomposed, with the formation of iodine chloride, which then reacts with the ammonia. The nitrogen iodide is soluble in an excess of either of the reagents, and it is specially necessary to avoid adding too much potassium iodide. No black coloration that could be confused with that given by ammonia is produced by amines, amides, ureides, pyridine derivatives, nitrates or nitrites. Methylamine gives a red coloration, appearing blue by transmitted light, and aniline a reddish-brown colour, both of which can readily be distinguished from the colour produced by ammonia. On the other hand, the nitrogen iodide reaction is given by all ammonium salts, including the cyanide and sulphide, the presence of which interfere with Nessler's reaction.

Drinking-Water.—From 20 to 30 c.c. of the undistilled water are treated with 3 drops of a 10 per cent. solution of potassium iodide, and 2 drops of a concentrated solution of alkali hypochlorite (commercial *eau de Javelle*), and there is an immediate turbidity or brownish-black precipitate when the quantity of ammonia exceeds 2 mgms. per litre of water. In the case of smaller quantities the water is evaporated in the presence of a very small amount of sulphuric acid, which is roughly neutralized before applying the test. The small quantity of iodine liberated in the reaction gives a slight yellow tint to the water, which does not interfere with the test. In doubtful cases, however, a comparative blank test with pure water may be made. A *very slight* excess of the hypochlorite solution destroys the iodine coloration without affecting that of the nitrogen iodide. The iodine may also be separated from the iodide by means of chloroform, but as a rule these precautions are unnecessary. A colorimetric determination can be made in the same way as in Nessler's process. A table of comparative results obtained with different drinking-waters shows that the new method gives figures in close agreement with those obtained by Schloesing's method. C. A. M.

On the Quantitative Determination of Iodine in Soluble Iodides and in Mixtures of Iodides with Bromides and Chlorides. Hugo Ditz and B. M. Margosches. (*Chem. Zeit.*, 1904, xxviii., 1191.)—The authors determine iodine in soluble iodides by decomposing them with potassium iodate in the presence of a small excess of dilute sulphuric acid. The liberated iodine may be shaken out with toluene, the iodine being either titrated directly in the toluene solution, or the excess of iodate in the aqueous solution may be determined by adding an excess of iodide and titrating the liberated iodine. Or, again, the iodine may be removed from the solution by boiling, and the excess of iodate determined as above. Any one of these modifications may be used to determine iodides in the presence of chlorides. When bromides are also present, however, the boiling-off method may not be used, as bromides are attacked in the heat by iodate in acid solution, even when acetic acid is substituted for sulphuric acid; and even working in the cold, the excess of sulphuric acid should be as small as possible, the amount necessary being best determined by a preliminary experiment. Acetic acid, however, may be used in fair excess in the cold. The results obtained in a large number of test analyses by the above methods were good, the error generally being less than 1 mgm. of iodine on quantities of iodine up to 0·5 gram. A. G. L.

Determination of Small Quantities of Bromine and Chlorine in Iodine. R. R. Tatlock and R. T. Thomson. (*Journ. Soc. Chem. Ind.*, 1905, xxiv. 187.)—Five to 10 grams of commercial iodine are treated with 50 to 100 c.c. of water, and finely-granulated zinc or zinc dust carefully added in small quantities at a time with frequent agitation, until all the iodine is converted into zinc iodide. The temperature should not sensibly rise during the operation. The solution is filtered, and 3·5 to 7 grams of sodium nitrite (according to the amount of sample originally taken) added to the filtrate. The liquid is acidified (not strongly) by the cautious addition of *diluted* sulphuric acid, when all the iodine is liberated, but no bromine is set free if diluted sulphuric acid is used. The precipitated iodine is filtered off, washed with a little cold water, and the filtrate shaken with benzene, chloroform, or carbon disulphide, to remove any free iodine in solution. The aqueous liquid is separated and again treated with sodium nitrite and acid, and shaken out with benzene.

Any traces of the solvent may next be expelled by heating the liquid, and the bromine and chlorine then precipitated by the addition of excess of silver nitrate and some nitric acid, and the liquid boiled. The silver salts are collected on a tared filter, and thoroughly washed with hot water. The silver chloride is then dissolved out of the mixture by repeatedly pouring through the filter about 60 c.c. of a solution prepared by dissolving 2 grams of silver nitrate in 90 c.c. of water, and adding 10 c.c. of ammonia (specific gravity 0·880), and the residue finally washed with the remaining 40 c.c. of the ammoniacal silver nitrate. The chloride is completely dissolved in this way, leaving the insoluble silver bromide, which is washed with warm dilute nitric acid, and then with hot water, dried, and weighed. The ammoniacal solution containing the silver chloride is acidified with nitric acid, heated, and the silver chloride collected as usual.

To test for iodide in the bromide precipitate, as should generally be done, it is treated with dilute sulphuric acid and zinc-dust until decomposition is complete. The liquid is then filtered and a limited quantity of chlorine-water added, and extracted with chloroform. In the presence of iodine a violet tint will be obtained, but when bromine only is present the chloroform has a reddish-brown colour.

A. R. T.

The Detection of Bromine in the Presence of a Large Quantity of Iodine. H. Cormimbœuf. (*Ann. de Chim. anal.*, 1905, x., 145-146.)—A solution of the iodides (or of hydriodic acid neutralized with sodium hydroxide), which must be neutral or very slightly acid, is treated with excess of a solution of ferric chloride (specific gravity 1·45), which precipitates the iodine in the form of a black powder. This is separated by filtration through cotton-wool, and the filtrate boiled until violet vapours no longer appear, in order to eliminate the last traces of iodine. The iron is then precipitated by means of sodium hydroxide, and the colourless filtrate from this precipitate is treated with a crystal of potassium chlorate and chloroform, followed by several drops of concentrated sulphuric acid. Any bromine present is liberated and taken up by the chloroform, which becomes more or less yellow. In this way 0·1 per cent. of bromine can easily be detected.

For the detection of bromine in commercial iodine several grams of the sample are treated with an excess of reduced iron in the presence of 50 c.c. of water. The ferrous iodide formed under these conditions will contain ferrous bromide if bromine is present. The liquid is filtered from the undissolved iron, and the filtrate treated with ferric chloride, etc., as above described.

C. A. M.

The Precipitation of Barium Bromide by Hydrogen Bromide. Normann C. Thorne. (*Zeits. anorg. Chem.*, 1905, xliii., 308.)—The author shows that barium is completely precipitated as bromide when barium bromide is dissolved in a small quantity of water and a mixture of ether and of a saturated aqueous solution of hydrogen bromide is added, especially readily if the solution be then saturated with gaseous hydrogen bromide. The presence of ether is not absolutely necessary. Barium may be separated in this way from considerable quantities of calcium and magnesium. On drying the precipitate, a small quantity of oxybromide is formed; this may be obviated by previously washing the precipitate with a solution of ammonium bromide, and drying first at a low temperature and then at 250° C. until the ammonium bromide is completely volatilized.

Hydrogen bromide will also precipitate barium completely as bromide from concentrated solutions of the chloride. Hydrogen chloride throws down the chloride quantitatively from these solutions. When both hydrogen chloride and hydrogen bromide in considerable quantity are added, the precipitate consists of a mixture of barium chloride and bromide.

A. G. L.

Diphenylamine as a Reagent for Nitrites, Nitrates, and Chlorates, and its Use when mixed with Resorcinol and β-Naphthol. E. P. Alvarez. (*Chem. News*, 1905, xci., 155.)—The author employs a sulphuric acid solution of mixtures of diphenylamine with resorcinol or β-naphthol, as the colorations obtained are more persistent and are more easily distinguished from one another than when diphenylamine is used alone. The reagent is prepared by dissolving 0·1 gram each of diphenylamine and re-sublimed resorcinol in 10 c.c. of sulphuric acid. Five or six drops of this solution are poured on about 1 mgm. of the salt to be tested. With *nitrates*, a very permanent yellowish green coloration is obtained, the edges of the spot becoming blue on spreading the liquid out. On adding alcohol, an orange-coloured solution is obtained. With *nitrites*, a deep blue-violet colour is yielded. On moving the liquid, the edges of the spot become red, and a red solution is obtained on the addition of alcohol. With *chlorates*, the result is not satisfactory, but by substituting β-naphthol for the resorcinol, a dull-green coloration is obtained, which, after a time, changes to a deep grey, almost black. The solution resulting from the addition of alcohol is greyish or blackish. W. P. S.

Some Reactions of Hydrofluosilicic Acid. A. Gawalowski. (*Zeit. anal. Chem.*, 1905, xliv., 191-194.)—An aqueous solution of hydrofluosilicic acid of 1·06 specific gravity gives characteristic reactions with 10 per cent. solutions of the following reagents: *Sulphuric acid* yields a white crystalline precipitate, which is insoluble in water, dilute nitric acid (1 : 10), and alcohol, but dissolves in potassium hydroxide solution, rapidly on heating, whilst large white flakes simultaneously separate. *Potassium chromate* gives a compact yellow precipitate, whilst the supernatant liquid is yellow at first and then colourless. The precipitate darkens in hot water. It is insoluble in cold nitric acid and alcohol, but dissolves in hot or cold potassium hydroxide solution and in sulphuric acid, with the simultaneous separation of flakes. *Potassium bichromate* gives an orange-yellow crystalline precipitate insoluble in water, cold nitric acid, cold potassium hydroxide solution, and alcohol, but soluble in traces in hot nitric acid and potassium hydroxide solution. *Hydrochloric acid* (specific gravity 1·10) gives a white compact crystalline precipitate, while the supernatant liquid is a faint bluish-green. It is slowly soluble in hot water and in cold nitric acid and potassium hydroxide solution (no separation of flakes). *Chromic acid* gives a yellow precipitate which is insoluble in the different reagents, with the exception of hot potassium hydroxide (separation of flakes). *Potassium bichromochloride* gives an orange precipitate insoluble in cold water, cold nitric acid, alcohol, and a cold mixture of alcohol and sulphuric acid, but very slightly soluble in hot nitric acid. Cold potassium hydroxide solution turns the precipitate yellow and then dissolves it (separation of flakes). A hot mixture of alcohol and sulphuric acid decomposes it, with the formation of a green colour. C. A. M.

Estimation of Phosphoric Acid. F. Raschig. (*Zeits. f. angew. Chem.*, 1905, xviii., 374.)—One of the chief desiderata in technical analytical chemistry is to replace tedious gravimetric methods by quick practicable volumetric processes. Quite recently G. v. Knorre has shown that it is possible to estimate sulphur in pyrites with equal accuracy and great gain in time, compared to the barium chloride method, by precipitating as benzidine sulphate, and estimating volumetrically its content of acid by $\frac{N}{10}$ soda.

The principle underlying this process is that, by washing a precipitate not absolutely insoluble with quantities of water so small as not to make the error due to the solubility appreciable, it is yet possible to free the precipitate from mother-liquor sufficiently to make error from that source negligible.

The author set himself to determine whether this principle could not be applied to other cases, and selected the estimation of phosphoric acid as being even more frequently required than that of sulphuric acid.

At present the method in general use is the gravimetric estimation as $Mg_2P_2O_7$, where the phosphoric acid is first precipitated as $MgNH_4PO_4$, a substance somewhat soluble in water, and therefore usually washed with dilute ammonia solution.

A. Hebebrand suggested the titration of this precipitate with carminic acid as indicator, but he washed with ammonia, and eliminated the ammonia with alcohol. This was somewhat tedious and inconvenient, and the method has not obtained general recognition.

The author, who has used the method of washing with small quantities of water, made some trial experiments in the following manner: Twelve lots of 20 c.c. each of a $\frac{N}{10}$ solution of sodium phosphate (=3·1 gram P per litre) were precipitated with 10 c.c. magnesia mixture, and the precipitate collected on a suction filter designed by the author (*Zeits. f. angew. Chem.*, 1903, xvi., 818). The filtrate was returned into the vessel used for the precipitation, shaken well round, and again poured on to the filter, so that the whole precipitate was transferred to the filter without using any water. The first was not washed at all, the other eleven with increasing amounts of water (see table below). Finally the filter and precipitate were placed in a wide-necked flask of about 200 c.c. capacity, a drop of methyl orange added, and titrated with $\frac{N}{10}$ hydrochloric acid. The reaction proceeded very rapidly, the precipitate dissolving quite easily off the paper. The colour change occurred when the salt $NH_4H_2PO_4$ was formed. The precipitate from 20 c.c. of the original sodium phosphate solution should require 40 c.c. $\frac{N}{10}$ HCl. The results obtained were:

Experiment.	Volume of Wash-water in C.C.	C.C. $\frac{N}{10}$ HCl.
1	0	45·1
2	1	42·6
3	2	41·7
4	3	40·6
5	4	40·0
6	5	40·0
7	10	40·0
8	20	39·8
9	40	39·6
10	80	39·2
11	160	38·7
12	320	37·2

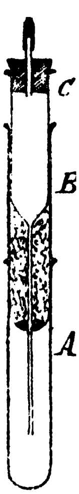
From these figures it appears that 4 c.c. sufficed to get rid of the alkaline mother-liquor sufficiently to avoid error, whilst the volume of the wash-water could be increased to 40 c.c. without introducing an error of more than 1 per cent.

The author suggests the following mode of procedure for the estimation: An amount of substance should be weighed out which will not contain more than 0·15 gram P_2O_5, requiring, therefore, less than 50 c.c. of $\frac{N}{10}$ acid. The solution is then precipitated in an Erlenmeyer flask with magnesia mixture, and filtered off with the aid of the suction-pump, using a filter-paper of 40 mm. diameter. The clear filtrate is used to rinse the rest of the precipitate on to the filter, and the mother-liquor is sucked off as completely as possible, 10 c.c. of water poured on to the filter and sucked through, then another 5 c.c., and the precipitate once more dried as far as possible by suction. The filter and precipitate are then transferred, with the precipitate upwards, by means of a pair of forceps to a beaker. The forceps and funnel are washed with the least possible amount of water, a drop of methyl orange added, excess of $\frac{N}{10}$ acid added, and the excess estimated by $\frac{N}{10}$ caustic soda.

E. K. H.

APPARATUS.

A Simple Apparatus for the Determination of Volatile Substances by Difference. L. Lehn Kreider. (*Zeits. anorg. Chem.*, 1905, xliv., 154.)—The apparatus shown consists of three tubes, of which *A* is an ordinary test-tube; *B* is a test-tube in the bottom of which a hole is made; and *C* is a test-tube drawn out to a long narrow tube at the lower end. The three tubes fit closely into each other as shown, the capillary of *C* going through the hole in *B*, in which it is packed with cotton-wool, the space above in *B* being filled with granular calcium chloride to dry the escaping gas. The space between *A* and *B* is made tight by means of a ring of melted paraffin wax. The substance to be acted on is placed in *A*; the liquid reagent (acid, hypobromite solution, etc.) is sucked up into *C* through the narrow tube and kept in the upper part by inserting a stopper carrying a glass tube closed by a piece of rubber and a glass rod. The whole apparatus is then put together and weighed. On removing the piece of glass rod from the top of *C* the liquid descends into *A*, acts on the substance, and the gas evolved, after being dried by the calcium chloride, escapes through the narrow annular space between *B* and *C*. A current of air is then led in at the top of *C* and the apparatus again weighed. A number of determinations made in this way of the carbon dioxide in calcium, barium, and strontium carbonates, of the hydrogen generated by magnesium and zinc, and of nitrogen in urea, ammonium oxalate, and ammonium chloride, show errors of, at the most, a few tenths of a mgm.

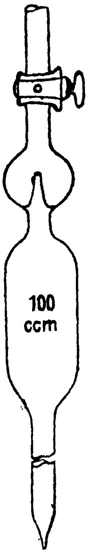


A. G. L.

On the Use of Vessels of Quartz Glass in the Laboratory. F. Mylius and A. Meusser. (*Zeits. anorg. Chem.*, 1905, xliv., 221.)—A number of tests made in the Reichsanstalt show that quartz vessels are not attacked by pure water, or by neutral or acid solutions. Concentrated sulphuric acid has a very slight action at 100° C.; phosphoric acid corrodes the quartz strongly above 400° C. Alkalies take up considerable quantities of silica. Saturated solutions of barium hydroxide also act on the quartz, but very slowly. From a 30 per cent. solution of caustic potash the vessels take up a small amount of potash, which cannot be washed out with cold water, but can be recovered by boiling with water. Soda does not appear to be absorbed in this way. Certain organic dyes—*e.g.*, methylene blue, congo red, rhodamine, aniline blue, and iodeosin, are also absorbed in very small quantities by vessels of quartz glass.

A. G. L.

On Density Determinations by Means of a Pipette and the Preparation of Volumetric Solutions according to Specific Gravity. F. W. Küster and Siegmar Münch. (*Zeits. anorg. Chem.*, 1905, xliii., 373.)—The authors show that with proper care the density, as determined by weighing the liquid delivered by a 100 c.c. pipette, is accurate to about 0·00005. The degree of accuracy may be still further increased by making use of the overflow pipette shown. This is filled by suction until the liquid overflows into the upper bulb, when the stopcock is closed; on opening the stopcock the same volume of liquid is always delivered. In filling and emptying the pipette the point should always dip into the liquid to the same depth (about 0·5 centimetre); the point should be finely drawn out; the temperature must be kept constant within 1° C.; and the pipette should be very frequently cleansed with a warm solution of potassium bichromate in sulphuric acid. Using this pipette, the error in the density should not exceed 0·000026.



The pipette may be used in conjunction with Table XIII. of Küster's "Logarithmische Rechentafeln für Chemiker" for the preparation of standard volumetric solutions, but the authors find that it is more convenient to find the weight of water delivered by the pipette at 18° C., and then always to work at this temperature. They give a table showing the number of c.c. of hydrochloric acid of various densities which must be diluted to 1,000 c.c. in order to prepare normal acid. The error in the strength of acid prepared in this way does not exceed 0·001 per cent. A. G. L.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, May 3, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. G. J. Alderton, B.Sc., Oscar Guttmann, J. T. Hewitt, M.A., D.Sc., Ph.D., A.R.C.Sc., and G. D. Lander, D.Sc., were read for the second time.

Messrs. E. R. Bolton, J. T. Dunn, D.Sc., E. K. Hanson, M.A., F. T. Harry, and F. R. Henley, M.A., were elected members of the Society.

The following papers were read: "Notes on Preservatives," by E. G. Clayton; "Notes on the History of Distilled Liquors—especially Whisky and Brandy" (with lantern illustrations), by Thomas Fairley; and a "Note on an Objectionable Method of Fining Wines," by R. Bodmer.

JULY, 1905. Vol. XXX., No. 352.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Charles Frederick Cross, B.Sc. (Lond.), F.I.C., 4, New Court, Lincoln's Inn, London, W.C., analytical and consulting chemist; and Charles Edgar Male, A.I.C., Brookland House, Cottenham, Cambridge, assistant to Mr. J. West Knights, were read for the first time.

Messrs. G. J. Alderton, Oscar Guttmann, J. T. Hewitt, and G. D. Lander were elected members of the Society.

The following papers were read: "The Separation of Strychnine and Brucine," by D. Lloyd Howard; "Ammonium Oxalate: its Formula and Stability," by P. V. Dupré; "Notes on some Abnormal Milks from Cleveland and South-East Durham," by A. C. Wilson; "A Simple and Convenient Camera for Photomicrographic Work," by A. C. Wilson; and "The Composition and Analysis of Milk," by H. Droop Richmond.

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ON THE ACTION OF SLIGHTLY ALKALINE WATERS ON IRON.

BY CECIL H. CRIBB, B.Sc. (LOND.), F.I.C., AND F. W. F. ARNAUD, F.I.C.

(*Read at the Meeting, April* 5, 1905.)

IT has been the practice of one of us during the last ten or fifteen years, when examining waters intended for use in steam boilers, to make a trial experiment with the water, using a slip of thin steel 4 inches long by 1 inch wide.

A hundred c.c. of the water are placed in a boiling-tube, the steel slip—brightly polished with emery, not cleaned by chemical means—inserted, and the whole kept at 100° C. (or as near to that temperature as a water-bath will bring it) for twenty-four hours, after which the nature of the action is examined and its extent measured. In most cases a similar experiment is made at the air temperature. At the end of the time the deposit, if any, is washed off the surface of the metal, and the total amount of iron in solution and in suspension in the water is estimated.

Of course the test is a very imperfect one. It does not reproduce in any way the conditions obtaining in a steam boiler; but some of those conditions, such as the high temperature and pressure, the rapid circulation, and the constant influx of fresh feed water, could not possibly be imitated without altogether prohibitive expense and trouble.

The plan has obvious advantages. It is simple and cheap; requires but little of the water; the conditions are under control, and can always be exactly reproduced; any peculiarities in the action are visible, and its extent is easily and rapidly estimated.

In the somewhat limited number of cases in which such a method of testing is likely to be of use it has been found very fairly satisfactory. As a means of investigating the problems connected with the action of water upon iron and steel, there is a good deal to be said in its favour. The quantitative results obtained respond to almost every change in the conditions. They are affected by the chemical composition of the saline residue of the water, by its gaseous contents, by the temperature, by the duration of the experiment, by the concentration of the saline contents of the water, by the physical or chemical condition of the surface of the metal, by the electrical condition of the metal, and by the presence or absence of light. It is not unreasonable, therefore, to conclude that in the majority of cases in which a natural water has a corrosive action on the metal of a boiler, some indication will be given of the fact by this simple method of trial. In actual practice this conclusion has been fully borne out, though opportunities for comparison between the behaviour of the water in the laboratory and that in the boiler are not so very frequent, inasmuch as the majority of waters cause incrustation and do not corrode.

Having had occasion to apply the method to artificially softened waters, in which, as is almost invariably the case, the liquid was slightly alkaline owing to the presence of lime, caustic soda, or sodium carbonate, it was found that when the alkalinity exceeded a certain amount no action whatever occurred, and the surface of the metal was as bright at the end as at the beginning of the experiment. This inhibitive action is, we believe, generally known; but we have nowhere seen any recognition of the fact that it has its limits, below which action does occur, although the water is still alkaline, much less any statement as to what those limits are. There seems to be a widespread impression that alkaline waters do not act upon iron at all, and the fact is usually regarded as one of the great advantages of the chemical treatment of hard waters.

We find, however, that with an alkalinity not exceeding a certain amount—dependent on the nature of the alkali present, and to a less extent on the temperature—not only does action occur equal in intensity to what would occur in the absence of any alkali, but that this action takes place in a very curious and interesting manner, for which at present we are unable to offer any altogether satisfactory explanation.

The following table (I.) shows the effect of adding to New River water small proportions of sodium hydroxide, lime-water, and sodium carbonate. The quantities added are expressed in parts of the alkali or carbonate per 100,000 of water, and also in cubic centimetres of decinormal solution per 100 c.c., and the amount of the action in milligrammes of Fe_2O_3.

TABLE I.—TWENTY-FOUR-HOUR EXPERIMENTS WITH NEW RIVER WATER TO WHICH HAD BEEN ADDED SMALL QUANTITIES OF ALKALINE SALTS.

		AT 100° C		AT AIR TEMPERATURE	
C C. of $\frac{N}{10}$ Alkali in 100 c c of Water.	Alkali by Weight in 100,000 Vols. of Water.	Area and Nature of Action	Iron Removed, expressed as Milligrammes of Fe_2O_3	Area and Nature of Action.	Iron Removed. Mgs. of Fe_2O_3.
		Series 1.—Sodium Hydroxide.			
1·0	4·0	General.	23·0	General, but on one side only.	9·0
2·5	10·0	Local; large patches.	18·3	Broad ill-defined patches.	7·3
5·0	20·0	Local; smaller; well defined.	23·0	Projecting growths, well defined.	7·0
10·0	40·0	Ditto.	19·3	No action.	0
15·0	60·0	No action.	0	" "	0
		Series 2.—Calcium Hydroxide.			
0·5	1·85	General.	25·5	General.	9·0
1·25	4·6	Many small rounded patches.	25·3	Few rounded patches.	6·5
2·5	9·25	Long ill-defined streaks.	18·0	Streaks, ill defined.	7·0
5·0	18·5	Smaller well-defined patches.	21·0	Patches, well defined.	7·0
7·5	27·7	Round, sharply-defined growths.	18·5	Very little action.	7·0
10·0	37·0	Three small patches.	24·0	No action.	0
15·0	55·5	No action.	0	" "	0
		Series 3.—Sodium Carbonate.			
1·0	5·3	General.	22·5	General.	9·0
2·5	13·25	Many small spots.	21·3	General, but more pronounced in parts.	10·0
5·0	26·5	Well-defined streaks.	14·0	Long patches, ill defined.	7·5
10·0	53·0	Well-defined projecting patches.	15·0	Ill-defined patches.	5·0
15·0	79·5	Large ill-defined patches.	14·5	Well-defined patches.	7·0
20·0	106·0	Few narrow streaks, sharply defined.	20·2	Round, well-defined spots.	6·0
30·0	159·0	Few small spots and one narrow streak.	15·0	No action.	0
40·0	212·0	No action.	0	" "	0

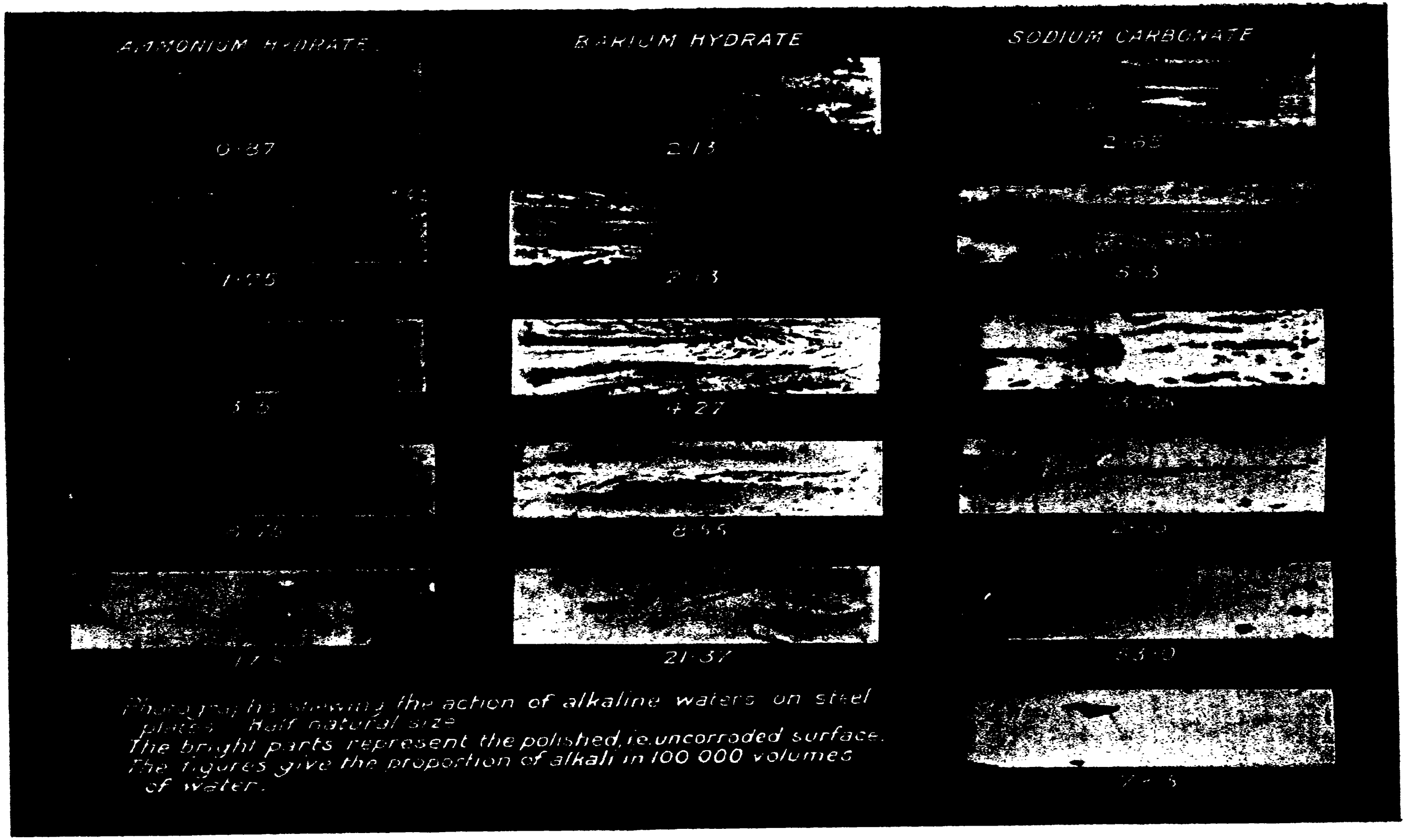
The peculiarity of the action consists in this—that whereas with quantities below those given in the table the iron slips are acted upon fairly uniformly all over, rust being formed on the surface of the plates, and then, owing to their nearly vertical position, settling slowly down to the bottom of the vessel; with larger but still very small proportions of alkali the action on the iron assumes a local character. At first the localization is extremely slight, but with increasing alkalinity not only do the areas acted upon get smaller, but they get to be more and more sharply defined; finally, when a certain strength of alkalinity is reached all action ceases, and at this stage no deposit is observable in the liquid, and the surface of the metal remains as bright and untarnished after twenty-four hours as at the beginning of the experiment, only a trace of iron being found in solution in the liquid, and none in suspension, after several days.

If in those cases in which action has taken place the amount of iron removed from the metal slips be estimated, it will be found to be—with the smaller proportions of alkali that produce any degree of localized action—not markedly different from that produced by the water alone. As the alkalinity increases the quantity diminishes slightly, but not at all in proportion to the area of action. Frequently when the area acted upon is not $\frac{1}{100}$ part of the total surface of the plate, it will be found that the iron removed has only diminished by about 5 per cent. of the maximum amount. When the higher limit of alkalinity is reached the quantity of iron removed diminishes with a sudden drop almost to nil—*i.e.*, to quantities which give no visible precipitate with ammonia. Coincident with the increased localization of the action is a tendency for the oxide of iron (or carbonate?) formed to adhere to the surface of the metal instead of slowly subsiding to the bottom of the liquid, and this tendency goes on increasing until the rust projects from the surface like the growth of a mould, and these growths get firmer and more closely adherent as they get fewer in numbers and smaller. In most cases they are of a dark-green or even blue colour beneath the surface, but if examined *in situ* without shaking they are generally found to be covered with a loose layer of bright-red oxide. A qualitative examination is sufficient to show that the greater part of the iron they contain is in the ferrous state, and it seems fairly certain that a ferrous salt is first produced, to be afterwards oxidized to ferric when it ceased to be in contact with the metallic surface. When rubbed off, the growths leave evidence of their action on the metal in the form of dark-coloured patches, which can be felt as distinct and generally sharply-defined depressions on the surface of the plates.

For the purpose of studying the phenomena under somewhat simpler conditions, a number of experiments have been made with distilled water instead of New River water. The dissolved salts in the latter are more or less precipitated by the alkaline hydroxides and salts which formed the subject of the experiments recorded in Table I., and the dissolved gases are greater and more variable in amount, so that the alkali added does not represent the alkalinity of the solutions. With distilled water the dissolved gases are the only uncontrolled constituents.

We have determined, for all the ordinary alkaline hydroxides and carbonates used, or likely to be used, in water softening, the degree of alkalinity at which this local action begins and finishes, and we have noted certain points in connection with the nature of the action, which appears to differ both in kind and in intensity with different bases (see photographs).

Table II. shows the results of experiments made in the cold, and at, or just below, the boiling-point, with potash, soda, ammonia, lime, and baryta; and Table III. with sodium carbonate.



Photographs showing the action of alkaline waters on steel plates. Half natural size.
The bright parts represent the polished, i.e. uncorroded surface.
The figures give the proportion of alkali in 100 000 volumes of water.

[*To face p.* 228.

TABLE II.—TWENTY-FOUR-HOUR EXPERIMENTS WITH DISTILLED WATER TO WHICH ALKALINE HYDROXIDES HAD BEEN ADDED.

At 100° C.				At Air Temperature.	
C.C. of $\frac{N}{10}$ Solution per 100 c.c. of Water.	Alkali by Weight in 100,000 Vols. of Water.	Area and Nature of Action.	Iron Removed, expressed as Milligrammes of Fe_2O_3.	Area and Nature of Action.	Iron Removed. Mgs. of Fe_2O_3.
		Series 1.—Sodium Hydroxide.			
0·25	1	Liquid turbid; action general, but slightly localized, in streaks.	20·3	Ill-defined streaks one side; general the other.	5·3
0·50	2	Liquid turbid; action general, but more pronounced in parts.	14·5	Fairly general, tending to streaks.	6·7
1·0	4	Long narrow patches.	18·5	Distinctly local; irregular patches.	7·0
2·5	10	Narrow patches.	19·3	One small projecting growth.	2·8
5·0	20	Two small streaks.	5·8	No action.	0
10·0	40	No action.	0		0
15·0	60	,, ,,	0		0
		Series 2.—Potassium Hydroxide.			
0·25	1·4	Long thin streaks over whole plate.	17·8	General, but tendency to streaks.	5·0
0·5	2·8	Thin streaks, less than above.	13·7	Long thick streaks, well defined.	6·2
1·0	5·6	Thin streaks, still less.	13·7	Few short thin streaks.	5·8
2·5	14·0	One large patch.	12·8	No action.	0
5·0	28·0	No action.	0		0
10·0	56·0	,, ,,	0		0
15·0	84·0	,, ,,	0		0
		Series 3.—Calcium Hydroxide.			
0·125	0·46	General on one side; patchy the other.	19·0	General, but tending to streaks on one side.	7·5
0·25	0·92	In narrow wavy streaks.	17·0	General, but more pronounced in parts.	8·5
0·50	1·85	In long patches.	20·8	Long patches.	6·5
1·25	4·6	In long streaks.	22·0	A very few patches.	9·5
2·5	9·25	Well-defined streaks.	20·5	,, ,, ,,	7·5
5·0	18·5	Very little action.	3·5	Little apparent action.	5·0
7·5	27·75	No action.	0	No action.	0

Series 4.—Barium Hydroxide.

0·25	2·13	Long narrow fern-like streaks.	15·7	General; one broad, ill-defined patch.	5·3
0·50	4·27	Broad, zebra-like markings.	20·0	Streaks and spots; form projecting growth.	7·3
1·0	8·55	Thick streaks projecting.	14·4	One thin streak.	5·2
2·5	21·37	One broad patch on one side; one spot on the other.	11·3	No action.	0
5·0	42·74	No action.	0	No action.	
10·0	85·48		0		
15·0	128·22		0	,, ,,	0

Series 5.—Ammonium Hydroxide.

0·25	0·85	Almost general, but some streaks.	19·7	General, but streaks just apparent.	7·5
0·50	1·7	Streaks along whole length.	17·0	Long patches, ill defined.	7·5
1·0	3·4	Well-defined streaks.	11·0	Long streaks, also spots.	7·0
2·5	8·5	Very thin streaks.	11·5	Few thin streaks and spots (projecting).	6·0
5·0	17·0	Few dots only.	11·5	Few spots.	5·0
10·0	34·0	No action.	0	No action.	0
15·0	51·0		0		

TABLE III.—TWENTY-FOUR-HOUR EXPERIMENTS WITH DISTILLED WATER TO WHICH SODIUM CARBONATE HAD BEEN ADDED.

At 100° C.				At Air Temperature.	
C.C. of $\frac{N}{10}$ Solution per 100 c.c. of Water.	Weight of Salt in 100,000 Vols. of Water.	Extent and Nature of Action.	Iron Removed, expressed as Milligrammes of Fe_2O_3.	Extent and Nature of Action.	Iron Removed. Mgs. of Fe_2O_3.
0·25	1·32	General; darker streaks visible.	22·0	Ill-defined patches.	8·5
0·50	2·65	Many long thin streaks, bright in between.	15·0	Long, broad patches.	7·5
1·0	5·3	Long patches.	16·3	Long patches.	7·0
2·5	13·25	,, ,,	14·0	,, ,,	5·5
5·0	26·5	Few broad patches.	14·5	Streaks.	6·0
10·0	53·0	Broad patches of smaller area.	16·5	Large well-defined spots.	6·5
15·0	79·5	Few patches.	17·5	Very sharply-defined spots.	7·0
20·0	106·0	Broad patch one side, one spot on the other.	16·5	Few spots.	6·0
30·0	159·0	Long broad patch one side, spots on the other.	18·5	No action.	0
40·0	212·0	No action.	0		
50·0	265·0	,, ,,	0		

Though each alkali has a more or less marked individuality as regards the pattern formed on the slips (shown fairly well in the photographs), and even the colour and tenacity of the growths of oxide, the same sequence of changes occurs with each as the proportion of alkali rises.

The figures in the tables show that the amount of iron removed is practically independent of the area attacked, so that the more restricted the area, the deeper the excavation that occurs there.

We have not yet made any definite experiments on the point, but from one or two isolated observations it would seem probable that, as the area of action gets less and the products of the action assume the form of growth-like projections from the metal, the proportion of ferrous iron increases. At all events, with a water of neutral reaction we could get no evidence of the presence of ferrous iron at all, while with the projecting "growths" ferrous iron was in greater quantity than ferric.

The colour of the oxide supports this view, inasmuch as the rust from neutral distilled water is always reddish-brown, while with the dilute alkaline solutions, and more especially with baryta, it is of a dark blue-green colour. In one case a growth of a light blue colour was observed—a not very surprising result, inasmuch as Nicolardot (*Journ. Chem. Soc.*, 1905) has shown that a white Fe_2O_3 can be prepared.

The fact that alkaline solutions are capable of acting in the way described is of considerable theoretical interest, as well as of possible practical importance.

To deal with the purely scientific aspect of the matter, it is necessary to consider the explanations that are usually given of the rusting of iron. According to the view originally proposed by Traube (*Berichte*, xviii., 1877-1887), and more recently advocated by Dunstan (*Proc. Chem. Soc.*, 1903, p. 150), oxygen and water react with the metal, *both* being *necessary*, and ferrous hydrate and hydroxyl are formed. The latter reacts again with iron, forming a hydroxide of the formula $Fe_2O_2(OH)_2$, which, according to Dunstan, represents the ordinary composition of iron rust. Neither of these workers has been able to detect the presence of hydrogen peroxide, but this is (to them) explained by the rapidity with which that substance is acted upon and destroyed by metallic iron.

Traube asserts, as a well-known fact, that iron is not oxidized when put into alkaline solutions, and states that it decomposes hydrogen peroxide when in such a solution without being itself oxidized. Moody more recently (*Proc. Chem. Soc.*, 1903, p. 240) has found that pure freshly-distilled hydrogen peroxide does not act on iron unless an acid, such as carbonic acid, is also present.

Dunstan (*loc. cit.*) explains this passivity of iron towards alkaline solutions by saying that all the substances which inhibit rusting do so by virtue of their power of decomposing the peroxide. If, therefore, dilute solutions of alkalies *do* act on iron, either hydrogen peroxide is formed, in spite of the presence of the alkali (in which case, according to Traube, it should not act on the iron), or the hydrogen peroxide is not formed, and the hydroxylation theory becomes untenable. It must be borne in mind, however, that Dunstan was dealing chiefly with the *atmospheric corrosion* of iron, and that he used apparently highly purified iron in his experiments.

To ascertain whether small quantities of hydroxyl could survive the action of the slight alkalinity of the solutions employed, we made up two solutions, containing respectively 0·15 and 0·7 parts of hydrogen peroxide per 100,000 of distilled water; 100 c.c. of each strength were heated in a water-bath for twelve hours, and two similar quantities, to which 1 c.c. of $\frac{N}{10}$ sodium hydroxide had been added, were treated in the same way. At the end of the time some of the original solutions which had not been heated and the four solutions that had been heated were tested for hydrogen peroxide, and abundant evidence of the presence of that substance—or, at all events, of a peroxide—was obtained in every case. The peroxide is not destroyed by even 5 per cent. soda after heating in the water-bath for four hours. From the way in which the experiments were carried out as little as 0·015 per 100,000 could have been detected. The action on iron (as measured by Fe_2O_3 removed) was not materially increased by the presence of the hydroxyl, and when the added alkali was raised to 15 c.c. it was entirely inhibited, just as if no peroxide had been present, although the latter could be easily detected at the close of the experiment (twenty-four hours).

It seems quite plain, therefore, that the hydrogen peroxide theory, as far as this particular form of rusting is concerned, cannot be sustained, unless as a mere pious belief unsupported by any sort of proof, other than the indirect one of analogy, for it cannot be proved to be present under ordinary circumstances, and when added it neither increases the action nor gives rise to it when it would otherwise not occur.

The other theory of the rusting of iron has its most recent exponent in Moody (*Proc. Chem. Soc.*, 1903, pp. 157 and 239), and is to the effect that the primary action is due to the interaction of iron and carbonic acid, and that rust is formed by the subsequent oxidation of ferrous salt. He shows that when iron is exposed to water and oxygen—previously freed as far as possible from carbon dioxide—the volume of oxygen remains practically unchanged, but that on admitting carbon dioxide it diminishes rapidly and the iron rusts.

It is as difficult to believe that carbonic acid is any more necessary to the reaction than hydrogen peroxide, in view of the experiments recorded in Table II., more especially those in Series 3 and 4. To make quite sure that the amount of carbonic acid gas dissolved in the distilled water was not greater than the quantity of baryta added, a special experiment was made in which 0·5 c.c. of baryta water was added to 100 c.c. of the water, and a steel slip immersed in the liquid. At the end of the twenty-four hours, when the usual characteristic action had taken place, titration of the clear solution, using phenolphthalein as indicator, showed alkalinity still present, corresponding to 0·4 c.c. $\frac{N}{10}$ $Ba(HO)_2$, a loss of only 0·1 c.c.

In view of the extreme insolubility of barium carbonate in hot water, it is not unfair to assume that neither free nor combined carbonic acid took part in the reaction.

Further, it would appear from another experiment that in the presence of what must be regarded as a large excess of free carbonic acid, iron is *not* acted upon, for we find that sodium bicarbonate acts in a similar way to the normal carbonate if only enough of the salt is present—*i.e.*, a stage is reached when the iron is entirely untouched after being immersed in the solution, either hot or cold, for twenty-four hours.

We have distinct evidence that localized action occurs with this salt, but in a far less striking manner than is the case with the *normal* carbonate. With 10 c.c. of a normal solution the action is general, but at the same time small; with 15 c.c. of normal bicarbonate solution per 100 of distilled water the plate remained absolutely bright and untarnished after being in the cold for twenty-four hours, and it gave no evidence (judging by the mercuric chloride test) of the presence of any normal carbonate at all—*i.e.*, the whole of the bicarbonate remained unchanged. A hot solution of the same strength was also without action, although much normal carbonate was formed—*i.e.*, a large amount of carbonic acid must have been present, or, at all events, must have passed out of the liquid.

Table IV.

C.C. of Bicarbonate (Normal Solution).	Bicarbonate per 100,000 Vols. of Water.	Mgms. of Iron Removed.		
	84	cold	8·5	action general.
		hot	12·5	,, ,,
5	420	hot	13·5	slight tendency to local action.
7·5	630	hot	12·5	
10	840	cold	7	ditto more marked.
		hot	8	
15	1,260	hot	0	

According to Moody, oxygen and water without carbonic (or other) acid will not cause rusting. According to Dunstan carbonic acid and water without oxygen is also without effect, while the various experiments we have made certainly indicate that carbonic acid is not necessary, though there can be no doubt that it plays an important part under the ordinary conditions of rust formation. Some further experiments go to show that the actual *quantity* of the dissolved gases has very little to do with the matter, for we find that after twelve hours at water-bath temperature no further gases are evolved from water; and yet when distilled water was first boiled and then heated for four hours on the water-bath in an open vessel, the action on iron after twenty-four hours was 25·3 mgms., as compared with 22·2 with the same water not previously heated. Similar experiments with water containing 2·5 c.c. $\frac{N}{10}$ NaHO gave, when heated beforehand, 13·2 mgms., and when not so heated 17·8. As water entirely free from gases will not act on iron, it seems probable that a minute quantity of gas is necessary to start the reaction, which then goes on continuously. It is obvious, however, that under the term "rusting" a number of different reactions are included, and much more work is required before a satisfactory explanation of them all is likely to be forthcoming.

It will, of course, at once be objected that the experiments were made with impure iron—in this case steel. We have not yet been able to get, in the form of slips, any iron of known purity, but we have found that sheet-iron and Siemens steel and also steel wire alike give the characteristic action within the limits of alkalinity we have established.

In summing up the result of his work (*loc. cit.*), Dunstan makes the statement (whether as the result of experiment or as a commonly-accepted opinion is not clear) that when the iron is impure, or when another metal is present, electrolytic action occurs; the electro-positive metal suffers oxidation and hydrogen gas is evolved. If the latter is a necessary condition of electrolysis, then no electrolysis takes place in connection with the phenomena under discussion; for if a boiling tube, prolonged at what would be its closed end by a narrow tube of $\frac{1}{16}$ inch internal diameter and about 4 inches long, closed at its distal extremity, be inverted over a steel slip immersed in water, of slightly alkaline or neutral reaction, and heated in the water-bath for twenty-four hours, the gas that collects in the narrow portion of the tube does not differ in volume by $\frac{1}{50}$ c.c. from that given off by the water alone in the absence of the steel slip.

To turn to the practical aspect of the question, it is quite obvious that, assuming that these curious phenomena we describe really take place under the conditions which obtain in boilers, feed-water tanks, etc., they would constitute a very serious objection to the use of chemical processes of water-softening, and also to the employment of alkaline waters of any sort for steam-raising purposes, for even the smallest degree of alkalinity in a feed-water will, by the concentration that takes place in the boiler, be raised to thirty or more times its original amount (Cribb, ANALYST, July, 1900), and therefore the whole range of alkalinity with which action occurs is well within the conditions that would occur in practice.

A glance at some of the plates is sufficient to show that even in the space of twenty-four hours (and especially where the area of attack is small) the metal is eaten away to quite an appreciable depth. Now, it is generally admitted that (leaving on one side the question of incrustation), the rusting action is a continuous one, although it slackens down considerably after the first onset, and the following figures (Table V.) show that this is so, and therefore, either hot or cold, it is quite plain that, given the right strength of solution, most serious pitting *might* be caused.

TABLE V.—ACTION OF DISTILLED AND NEW RIVER WATER DURING VARIOUS PERIODS AT THE AIR TEMPERATURE.

Period.	Distilled Water. Mgms. of Fe_2O_3.	New River Water. Mgms. of Fe_2O_3.
12 hours.	6·5	
24 "	8·0	9·1
48 "	—	14·4
168 "	25·8	—
240 "	39·5	56·0

It is important, therefore, to inquire how far, if at all, this could take place in practice.

As regards the metal, we find that sheet-iron, Siemens-Martin steel with the "skin" on, as actually used for making boilers, and the steel with which most of the experiments in connection with the paper were made, act practically alike. Even the "skin" does not prevent the local action in any way, and the action goes on at the same rate, or even more energetically, therefore the nature of the metal is no bar to the action.

The action of the dissolved salts likely to be met with in natural waters other than alkaline salts is similarly without any inhibiting effect.

As shown in Table VI., the figures represent mgms. of Fe_2O_3 removed from the steel plates after immersion in solutions of the salts named, and of the strengths specified, for twenty-four hours at the water-bath temperature.

TABLE VI.

Strength of Solution.	10 Parts per 100,000.	100 Parts per 100,000.
Sodium chloride	19·0	25·0
Sodium sulphate ..	18·5	28·0
Sodium nitrate ..	18·0	61·5
Sodium phosphate	18·0	16·5
Potassium nitrate	22·0	—
Calcium chloride ...	17·0	31·5
Calcium sulphate ...	25·5	54·5
Magnesium chloride	28·0	23·5
Magnesium sulphate ...	25·5	26·5
Ammonium chloride ...	12·0	47·5
Ammonium carbonate ...	30·0	22·0
Distilled water alone	19·0	22·0
New River water alone	25·0	30·0

Of course, only the salts of the alkalies and alkaline earths could be present under the conditions which give rise to local action.

In the case of boilers, especially high-pressure boilers, the high temperature prevailing increases the amount of decomposition undergone by practically all the saline constituents of natural waters (Cribb, ANALYST, 1900), and, therefore, it would naturally be expected that the possibility of pitting would increase rather than diminish as the temperature rises. To ascertain whether the local action of alkalies actually does take place at temperatures above 100° C., we have so far only made one experiment. It consisted in heating a steel slip immersed in distilled water, containing 4 parts per 100,000 of sodium hydroxide, to a temperature of 110° C. for some hours. The usual action characteristic of that degree of alkalinity took place. We hope, however, to have the opportunity before long of employing a still higher temperature; but there is no reason for doubting that the action at temperatures above 100° C. will be more, and probably considerably more, than it is at the normal boiling-point.

Even if the higher temperatures occurring in steam boilers brought about no increase in action, it is quite obvious that the rate of corrosion occurring in our laboratory experiments might have the most serious consequences even after a comparatively short period of working. Take, for instance, a 100 horse-power boiler with 600 square feet of water surface. Assume that it is fed with New River water, which according to Table VI. removes a little over 25 mgms. of Fe_2O_3 in twenty-four hours. This would mean 6·7 ounces of metallic iron removed in a single day and night. Each square inch of ½-inch boiler plate only weighs 1·22 ounces.

There are, on the other hand, some facts which would tend to lessen, if not altogether to prevent, pitting in a boiler, and there can be no doubt that these are largely operative, otherwise pitting would be universal with all softened waters from the cause under discussion.

The first is that most softened waters give a deposit when heated, which is generally sufficient to cover the plates and tubes with a more or less protective coating. If it is not, steps are taken to produce such a coating by what may be called artificial means. Secondly, the rapid circulation, especially in the case of tubular boilers, would almost certainly prevent anything in the nature of the growths seen in the laboratory experiments. There are, however, in most boilers, and especially in the non-tubular kinds, certain places where the circulation is very much slower, and it is in the experience of most engineers that it is in these spots that pitting most frequently occurs.

It would be unnecessary to refer to this point at such length but for the fact that in using the steel slips we have found that when local action has started in certain areas, and the growths attached are rubbed off the surface of the slip, action does not always commence again in the same place. When the slip was polished with emery before replacing it in the liquid, the action never commenced at the same spots.

Finally, there is another very interesting reason why action is less likely to occur in boilers and closed vessels, and that is that it is unquestionably less energetic in the dark.

The figures we bring forward in support of this are neither so numerous nor so striking as we should like, but we have never made a single experiment in which the stimulating effect of light is not apparent—that is to say, in which the amount of corrosion has not been less in the dark than in the light.

The following table shows side by side the result of exposing the same kind of steel to the action of the same liquid for the same time in ordinary daylight and in absolute darkness. The figures represent as usual mgms. of Fe_2O_3 removed.

TABLE VII.

	Duration.	In the Dark.	In the Light.
Distilled water	12 hours	4·5	6·5
Tap-water	24 hours	7·5	13·5
Distilled water, $MgCl_2$, 20 per 100,000 ...	„	5·2	7·2
„ „ $NaHCO_3$, 84 ...	„	6·0	8·5
„ „ Na_2CO_3, 53 ...	„	5·0	7·0
„ „ $Ca(HO)_2$, 9·2 ...	„	7·2	11·5
NaHO, 4	12 hours	2·0	4·2
Distilled water	240 hours	9·5	39·5
New River water	„	21·7	56·0
Distilled water + 4NaHO per 100,000	360 hours	16·0	48·5

The difference is considerably greater than would appear from the above, inasmuch as during somewhat less than half the total duration of the experiments (except, of course, the first) all the liquids were necessarily in the dark.

The further investigations we propose to undertake will, we hope, throw more light on this interesting point.

DISCUSSION.

The PRESIDENT (Mr. Bevan) said he should like to know more definitely how the authors accounted for the corrosion taking place in patches, except on the electrolytic theory, which, although their experiments seemed conclusive, he did not regard as being entirely disposed of. It would be interesting to know the actual composition of the iron, because, if the iron were chemically pure, it would be difficult to see how the action could take place in patches; while, if it were not chemically pure, it was pretty obvious that in certain places there might be a little more or a little less of one constituent or another, which, of course, would give rise to electrolytic action. In the case in which the corrosion took place on one side only, was the plate exactly vertical? Possibly the degree of exposure to light had some influence.

Mr. CRIBB said that he did not mean to imply that the action took place necessarily on one side only. It was curious, however, that this did sometimes happen. He did not think the difference with regard to light would be sufficient to account for it.

Mr. ARCHBUTT thought that it would have been better if the authors had started with steel boiler plate of known analytical composition, and had used strips from the same plate throughout the experiments. He also thought that it would have been better if the water had been freed from gases, as would be the case in a steam boiler. Although the authors had found corrosion to be so general in their experiments with alkali, whether in the caustic form or in the form of carbonate, he did not think that this was the case in actual practice, for, if it were, one would expect the boilers in the country generally to be in a much worse condition than they really were in. In certain cases, however, serious corrosion did occur from causes which at present could not be explained, and therefore any investigation which would help to throw light on these obscure causes would be useful. In a case within his knowledge in which pitting had occurred in boilers using Manchester water it had been remedied to a large extent by scraping out the magnetic oxide that had been formed in the pits, brushing these places over with petroleum, and using in the boilers about 1 pound of sodium carbonate per 1,000 gallons of water evaporated. The fact that this practically stopped the corrosion seemed to indicate that the action of the alkali in a boiler was not quite the same as under the conditions of the author's experiments. It might be mentioned that pitting and corrosion were nearly always local, pitting being sometimes so serious in boiler tubes as to result in holes right through the tubes. As a rule, in boiler plates also corrosion took the form of local pitting. He had at present under observation, however, an extraordinary case in which the barrels of locomotive boilers were corroded all over below the water-line. The boilers took water from several different sources, and all of these had been investigated, but no reason for the corrosion was at first apparent. No mineral or fatty acid could be traced as the cause. None of the waters were artificially softened, and no chemical or disincrustant was used in the boilers. The water of which the largest quantity was used came out of the Derbyshire limestone, and contained about 21 grains per gallon of total dissolved matter, including 7·55 grains of calcium carbonate, 3·99 grains of magnesium carbonate, 1·94 grains of magnesium sulphate, 4·74 grains of sodium sulphate, 1·19 grains of sodium chloride, and 0·95 grain of silica. The free carbon dioxide amounted to only 0·52 grain, and the dissolved oxygen to 0·66 grain. It would not be thought that such a water would seriously corrode boilers. He was, however, forced to conclude that such water was liable to corrode bare boiler plate, and that, unless the plates are protected by a scale, corrosion was more or less bound to occur. The scale which this water formed was particularly soft and porous, and did not protect the plates. It might be also that there was some organic acid in the water. Both of these causes would be met by softening the water with lime, which would remove the greater part of the carbonates but would leave the sulphates behind, thus encouraging the formation of a small quantity of thin, hard scale, whilst the excess of lime would neutralize any organic acid that might be present. An experiment on these lines now had been in progress for several months, and perhaps at some future time he might be able to communicate the result to the Society. Meantime he should be interested to hear if the authors could suggest any other explanation of the corrosion.

Mr. DIBDIN said that, in connection with the boilers at the pumping-stations of the London County Council, it was at first thought that magnesium chloride seemed to facilitate pitting as much as anything. This was particularly the case with some deep well waters, and especially with that from a trial well sunk at Crossness. The water there was brackish, and contained a good deal of magnesium chloride, derived, no doubt, from tidal water entering through a fault in the strata. The most effective preventive of this action on the boilers was found to be the use of an alkaline solution of tannin, made by boiling bark with caustic soda. The pitting seemed to take place with almost every kind of water, even when distilled in the ordinary process of condensation—New River water, tidal water, or water supplied, say, by the West Middlesex or East London Water Companies. He had often thought over the matter, but must confess that the only conclusion he had been able to arrive at was that the corrosion was due entirely to electrolytic action, and that it would, therefore, vary with the character of the iron. It seemed to him that, if the authors' experiments could be made in the reverse direction—namely, by taking varying qualities of iron and water of constant composition—a good deal of light might be thrown on the subject.

Mr. JULIAN L. BAKER suggested that, bearing in mind the medium in which the protuberant growths were developed, these possibly consisted largely of an anaërobic form of mould or bacterial growth. He should like to hear whether any of the experiments had been made under aseptic conditions.

Mr. CRIBB said that in every case where these growths developed in the cold they also occurred in the corresponding experiment at 100° C.

Mr. JOHN WHITE said that a short time ago he had submitted to him a piece of an iron branch water-main 3 inches in diameter. It had been used for about six months for a fairly pure public supply, which, however, contained some vegetable matter. After that time the pipe had become so "made up" that a lead pencil would not pass through it. The only conclusion he could come to was that, as the water contained free oxygen and carbonic acid, oxide of iron was formed, which attached itself to the walls of the pipe, and this caused the deposition of organic matter as well. The deposit was not an ordinary incrustation; it contained about 60 per cent. of oxide of iron, the remainder being water and organic matter.

Mr. J. H. B. JENKINS inquired whether the authors stated in their paper the extent to which corrosion took place with New River water and distilled water respectively to which no alkali had been added. The addition of caustic soda to London water would form carbonate with the bicarbonate present in the water, so that at a certain stage there would be present only the normal carbonates of soda and lime. Perhaps the authors could indicate in the tables when that stage was passed, and caustic alkali could be assumed to be present in the water. He had noticed in examining polished steel sections microscopically that the sulphide dots or threads always found in commercial mild steels became nuclei for corrosion, by which the sulphide was decomposed. After rubbing such corroded sections afresh there would probably be no longer any sulphide spots left in the earlier positions, but others would be exposed at the new surface, so that the next points of corrosion would be expected to appear at fresh places. In connection with the experiments in the light and in the dark, he should like to hear whether the temperature was about the same in each case, because generally one would expect the temperature to be somewhat higher in the daytime.

Mr. CRIBB said that the results of experiments with New River and distilled water alone were given in Tables VI. and VII. He thought that the point when all the bicarbonate in the New River water was converted into carbonate would probably have been either reached or passed when 5 c.c. of $\frac{N}{10}$ alkali had been added. With regard to the experiments in light and darkness, various methods had been adopted to insure equal temperature or nearly so.

Either the dark experiment was made in an incubator at 20° C. (*i.e.*, was slightly warmer than the other) or the two cylinders were placed side by side, one being under a cardboard cover and the other exposed. He thought that the differences of temperature would in most cases be far too small to produce any appreciable effect on the results.

Mr. JENKINS, continuing, said that in the boilers of which he saw most—namely, locomotive boilers—the possibilities of galvanic action were undoubtedly great, because the fire-box inside the boiler was of copper. In nearly all cases, however, unless the water was quite soft, a protective scale was formed, which kept the water from contact with the metal surfaces. Very often they met with cases in which, if the scale had been broken or was not sufficiently hard to be impervious, galvanic action occurred. When pitting and corrosion took place, it was generally at those parts of a boiler where the circulation was least vigorous. He thought that this was due to the fact that in those places the gases which came out of solution when the water was heated remained in contact with the metal for the longest time. In a locomotive boiler this was generally below the level of the fire, near the foundation ring, or along the belly of the boiler.

Mr. Herbert E. Burgess suggested that the local character of the action might be accounted for by the presence in certain places of a thin coating of grease. If the plate happened to be touched with the fingers, the grease therefrom would form a protective coating, and he suggested that it would be well to wash the metal with chloroform before the experiments were made.

The President thought that the peculiar pattern of the corrosion was against Mr. Burgess's theory.

Mr. Cribb, in reply, said that he himself should be inclined to consider that these phenomena were due to electrolytic action were it not for the difficulties in the way of accepting that explanation. With a material like Siemens-Martin steel it must be due to the distribution of the different impurities (*i.e.*, substances other than iron) throughout the mass of the metal, and therefore, assuming that one alkali would have the same chemical action as another, there was no particular reason why different alkalies should produce different electrolytic effects; and therefore the patterns should be the same. They were, however, very different. As far as he knew, the distribution of the different constituents of steel was a microscopic and not a macroscopic matter, and therefore any patterns due to electrolysis might be expected to be microscopic and not macroscopic.

Mr. Jenkins remarked that lines of impurity, such as sulphide threads, would be found drawn out in the direction of rolling of the steel plate. This happened to be the same direction as the corrosion streaks developed in the author's experiments. Supposing that other strips were cut so that their long dimensions were *transverse* to the direction of rolling, would the direction of corrosion streaks be altered?

Mr. Cribb said he had noticed with Siemens-Martin steels in the case of which the "skin" was allowed to remain on the front and the sides had been cut through, the local action took place on the sides as well. There was no connection between the pattern on the sides and that on the front. That was one of the difficulties standing in the way of the electrolytic theory. Practically the same character of pattern was obtained with different pieces of metal when the same alkali was used. With regard to the analytical composition of the metal, many of these experiments had been made a long time ago, when testing the value of the method as a purely empirical process for getting at the corrosive powers of different waters. The slips of metal used, however, were in all cases of the same composition, and they might be able to analyse one later on. Mr. Dibdin's suggestion would be rather difficult to carry out, as it was not easy to get in a convenient form samples of iron of varying and known composition. They had, however, been able to show that with actual steel boiler-plate the same phenomena occurred. They did not, of course, contend that every time an alkaline water was used in a boiler this local action would necessarily take place, and they had pointed out the causes which would be likely to prevent it. It was curious that, in spite of the widely-accepted opinion as to the corrosive effect of magnesium chloride, there was scarcely any evidence in its favour except that derived from the analysis of boiler waters themselves. A 1 per cent. solution of magnesium chloride, when heated, had no more action on metal than plain distilled or tap water. As a matter of fact, it did not decompose when boiled. He admitted that it apparently did decompose under high pressure, but he had not been able to make any laboratory experiments under those conditions, except one or two conducted in an autoclave which, however, failed to yield any conclusive results. Mr. Jenkins's suggestion that the impurities formed nuclei which were removed by rubbing afforded a very reasonable and probable explanation. The rubbing, however, was very slight, and, moreover, the action, especially when it was beginning, was hardly of a nature suggesting the existence of nuclei,* inasmuch as it commenced in the form of patches without anything in the nature of a nucleus. It was only when the alkalinity was reaching its higher limits that small dots were produced. He did not think that grease could have had any influence in these experiments, because all the plates were systematically and carefully cleaned with emery, and were never touched afterwards except at the edges. The strength of the alkali, too, would in some cases be sufficient to saponify any slight traces of grease. He was afraid he could not suggest any explanation of the curious case of corrosion mentioned by Mr. Archbutt.

ADDENDUM.

Mr. Archbutt writes that, having by the courtesy of the authors been permitted to read the paper more carefully since the meeting, he does not feel convinced that the chief corrosive agent was not, after all, carbonic acid, the action of which ceased when the amount of alkali added was sufficient. The authors do not appear to have determined the amount of free CO_2 originally present in the water, and therefore it is impossible to say what relation the amount of alkali which was found to stop corrosion bore to the free CO_2. Is it not possible that in highly-dilute solutions bicarbonates may be mildly corrosive to iron and steel, and need the presence of a certain amount of free caustic or monocarbonated alkali to restrain their action?

In the experiment with baryta, the amount added was just enough to neutralize 1·1 parts of free CO_2 per 100,000 parts of distilled water. Supposing more than this were originally present in the water, some of the barium carbonate may have been held in solution, and in the subsequent titration have neutralized acid. Therefore it seems that the experiment, as described in the paper, does not necessarily prove that the baryta removed all the carbonic acid.

To prove their case conclusively, the authors would need to show that distilled water which has been freed from every trace of carbon dioxide and oxygen by boiling, and has been prevented from reabsorbing any of these gases, is more corrosive to iron when slightly alkaline than when no alkali is added.

Referring again to the case of locomotive boiler corrosion, which had formed the subject of some of his previous remarks, he had within the last few days had an opportunity of inspecting two locomotive boilers, one of which had been supplied during the last nine months exclusively with the water of which the analysis had been stated, and the other with the same water after softening with lime. The difference in the appearance of the two boilers was striking. The boiler which had used the untreated water was already extensively corroded, whilst the boiler which had used the treated water was in very good condition, being only pitted in a few places here and there. Both boilers at the commencement of the experiment were nearly new and quite free from corrosion.

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* Since reading the paper experiments have been made with steel wire under the microscope, and most distinct evidence of nuclei, round which the action seemed to centre, has been obtained.

Mr. Cribb, in answer to the first point in Mr. Archbutt's "addendum," writes that the solution in the experiment referred to was titrated directly, using phenolphthalein, which does not react with barium carbonate. Also that in Table II., Series 4, an experiment is recorded in which local action occurred, the barium hydroxide added being equivalent to no less than 5·5 parts of CO_2 per 100,000.

Water really free from CO_2 and O does not act on iron at all. The boilers referred to in the final paragraph obviously confirm the author's contention that local action and pitting may occur in actual practice when artificially softened waters have to be employed.

The action of the water of which Mr. Archbutt gave the analysis before treatment was probably due solely to the small proportion of dissolved gases. The water in boilers is always being reinforced with fresh feed-water, and consequently there is always enough gas in solution to cause action, either general or local. In this case there was not enough of the scale-forming compounds to form a protective coating.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Ammonia in Milk. W. N. Berg and H. C. Sherman. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 124).—The authors believe that the determination of ammonia affords a valuable indication of the extent of the decomposition of the proteids in milk, and have consequently elaborated a method for its determination. Distillation with even exceedingly dilute sodium carbonate under atmospheric pressure yields erroneous results, due to the breaking-down of the organic nitrogenous compounds during the boiling, and hence recourse was had to a modified Boussingault-Schaffer method (*Amer. Journ. Physiol.*, 1903, viii., 330). In this method 50 c.c. of the milk and 50 c.c. of methyl-alcohol are distilled with 10 grams of sodium chloride and 0·5 gram of sodium carbonate in a two-litre flask under reduced pressure. The flask is closed by a two-holed rubber stopper fitted with an inlet-tube, through which air may be admitted, and a distilling head connected to two absorption cylinders, placed in ice-water and connected with a suction-pump by an empty wash-bottle. In the cylinders are placed 25 c.c. of $\frac{N}{20}$ sulphuric acid diluted to 40 c.c. with water. Boiling is started by bringing the flask into a water-bath at a temperature of 60° to 65° C., after which the temperature may be allowed to fall a little. With a pressure of 50 millimetres boiling should continue at from 56° to 62° C. After distilling for fifteen minutes, boiling is stopped; the volume of the distillate is noted, and this is then diluted to 250 c.c., and titrated with $\frac{N}{20}$ sodium hydroxide, using congo red as indicator. As the presence of methyl-alcohol affects the end-point, a correction must be made for it. This varies with the quantity of alcohol distilled over: for 30 to 39 c.c. of distillate it amounts to 0·4 c.c. of $\frac{N}{20}$ alkali; for 40 to 49 c.c., to 0·5 c.c.; and for 50 to 60 c.c., to 0·6 c.c. The volume of distillate obtained is usually between 45 and 55 c.c. A number of check experiments on water and milk containing known quantities of ammonium chloride showed the method to give exceedingly accurate results, the error generally being less than 0·1 c.c. of $\frac{N}{20}$ alkali.

In applying the method, it was found that if the addition of sodium chloride, which is used to diminish the hydrolytic dissociation of the sodium carbonate, is omitted, the results for fresh milk remain practically unaffected, whilst those given by stale milk are considerably higher. It is suggested, therefore, that by making a distillation both with and without the addition of sodium chloride, an indication of the easily decomposable proteids, as well as of the ammonia present, may be obtained in the case of stale milk. A number of tests in which the above ammonia method was compared with the "alkalinity" method of Richards and Woodman ("Air, Water, and Food," p. 152), showed that after making certain corrections for the alkalinity figure, it would be brought into comparative agreement with the ammonia figure in the case of fresh milk, yet for stale milk the ammonia figure increased much more than did the alkalinity figure, and hence the first afforded a truer indication of the state of the milk.

Preliminary experiments on the development on standing of acidity and ammonia in milk showed that there is no necessary connection between the two, since one may increase at a much greater rate than the other. A. G. L.

On the Specific Rotation of Salts of Casein. J. H. Long. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 363.)—The specific rotation of very thoroughly purified casein from cow's milk when dissolved in different alkali solutions was determined. It was found that for 5 grams of dry casein 45 c.c. of $\frac{N}{10}$ alkali hydroxide were needed to give a solution neutral to phenolphthalein; but half this quantity of alkali sufficed to give a practically clear solution. All readings were made at 20° C., using 5 grams of dry casein dissolved in 100 c.c. of the alkali solution. The results obtained are given in the following table:

Alkali Hydroxide.	C.C. of $\frac{N}{10}$ Solution Used.	$[\alpha]_D$
Sodium hydroxide	45·0	-103·5°
,, ,, ...	22·5	- 95·2°
,, ,, ...	67·5	-107·6°
,, ,, ...	90·0	-111·8°
Potassium hydroxide	45·0	-104·4°
Lithium hydroxide	45·0	-100·8°
,, ,, ...	22·5	- 94·8°
Ammonium hydroxide	45·0	- 97·8°

In connection with these results, it may be noted that Béchamp (*Bull. Soc.*

Chim. [3], iv. 18; ii. 152) obtained for casein dissolved in water $[\alpha]_j = -117{\cdot}7^\circ$, is approximately equal to $[\alpha]_D = -115^\circ$. A. G. L.

The Baudoin Reaction in Human Milk. Dr. Engel. (*Chem. Zeit.*, 1905, xxvii., 363.)—As it has been clearly shown that in the case of cows and goats this reaction cannot always be obtained in the milk of animals which have been fed with sesamé oil cake and even with the oil itself, experiments have been made on the same lines with the milk of wet nurses.

After a dose of sesamé oil had been given in salad, the furfurol reaction could be observed in the milk within a short time. After six hours it disappeared, but returned after ten, was more or less distinct for another four or five, at the end of which it finally disappeared. These results confirm the uncertainty of the reaction as a test for the feeding. A. N. C.

The Analytical Values of Goat's Butter. L. Hardy. (*Bull. Soc. Chim. Belg.*, 1905, xix., 13.)—Two samples of goat's butter examined by the author gave the following results, who calls attention to the very low Reichert value of this butter:

	Specific Gravity at 100° C.	Butyro-refractometer Reading at 40° C.	Crismer's Value.	Insoluble Fatty Acids per Cent.	Reichert-Meissl Value.
1.	0·8659	42	50	87·46	20·6
2.	0·8650	42	52·5	87·77	21·4

C. A. M.

Detection of Artificial Colouring Matters in Fats. H. Sprinkmeyer and H. Wagner. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 598, 599.)—Ten c.c. of the melted fat are placed in a separating funnel and dissolved in 10 c.c. of light petroleum. The solution is then shaken with 15 c.c. of glacial acetic acid. The presence of an artificial colour is shown by the lower—acetic acid—layer becoming yellow or red in colour, the latter coloration being given by alizarine and aniline yellows and by tropeolin. The ordinary colours employed for dyeing butter and margarine, such as Orleans, saffron, aniline, and alizarine yellows, turmeric, tropeolin, picric acid, and commercial butter colours, can all be detected by this method. W. P. S.

Examination of Lard from Pigs fed on Cottonseed Meal. L. M. Tolman. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 589-596.)—Working on a large number of samples of lard obtained from all parts of the bodies of pigs fed on cottonseed meal, the author was unable to detect the presence of phytosterol, even in those samples which gave a coloration with Halphen's test corresponding with an admixture of 15 per cent. of cotton oil (see ANALYST, 1901, xxvi., 292). It was found that the addition of 2 to 3 per cent. of cottonseed oil to lard could be detected by means of the phytosterol acetate method, and that heated cottonseed oil which does not give Halphen's reaction can be detected by this method. A. D. Emmett and H. S. Grindley, who also investigated this subject (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 263-270), found that these lards, besides giving a coloration with

Halphen's test, yielded crystals resembling phytosterol, showing that at least a part of the oil existing in cottonseed meal is absorbed by the animal body and transmitted in an unaltered condition to the fat cells. Tolman considers that a very large percentage of phytosterol must have been present in the residues obtained by Emmett and Grindley, as a mixture of 25 parts of phytosterol with 75 parts of cholesterol still gives the peculiar telescopic form of crystals mainly due to cholesterol. Before crystals are obtained which cannot be distinguished from phytosterol, at least 50 per cent. of the latter must be present. With such an amount present the acetate must have had a melting-point of 123° to 125° C., but this melting-point Emmett and Grindley were unable to obtain. W. P. S.

The Composition of American "Noodles" (Nudeln) and Methods for the Analysis of the Same. A. L. Winton and E. M. Bailey. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 137-142).—Egg-noodles are prepared from flour, with the addition of eggs or egg-yolks and salt. As manufacturers have recently placed on the market products dyed a golden yellow colour and devoid of eggs, the authors have examined a number of samples by Juckenack's method, with the object of ascertaining whether the presence or absence of eggs could be determined analytically.

Juckenack's method (*Zeit. Untersuch. Nahr. Genussm.*, 1900, iii., 13) is based on the determination of the lecithin-phosphoric acid in the sample, the details being as follows: Thirty grams of the finely-ground material are extracted for ten hours with absolute alcohol in a Soxhlet apparatus at a temperature, inside the extractor, of not less than 55° to 60° C. When the extraction is completed, 5 c.c. of 4 per cent. alcoholic potash are added to the contents of the extraction flask, and the alcohol is distilled off. The residue is transferred to a platinum basin by means of hot water, evaporated, dried, and charred. The charred mass is extracted with nitric acid, washed, burnt to a white ash, again extracted with nitric acid, and the phosphoric acid determined in the united solutions as usual. A determination of the amount of fat in the original sample is also of importance, as will be seen from the following table compiled from figures given by Juckenack and Pasternack (*Zeit. Untersuch. Nahr. Genussm.*, 1904, viii., 94):

Number of Eggs per Pound of Flour.*	Ash. Per Cent.	Total Phosphoric Acid. Per Cent.	Lecithin-phosphoric Acid. Per Cent.	Ether Extract. Per Cent.	Nitrogen × 6·25. Per Cent.
0	0·460	0·2300	0·0225	0·66	12·00
1	0·565	0·2716	0·0513	1·56	12·99
2	0·664	0·3110	0·0786	2·42	13·92
3	0·758	0·3482	0·1044	3·24	14·81
12	1·426	0·6123	0·2875	7·94	21·09

Only five samples contained appreciable amounts of eggs; and but two contained more than one egg, or the yolk of one egg, per pound of flour.* W. P. S.

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* The German pound here referred to is approximately 468 grams. All the results are calculated on the dry material.

Sophistication of Pimento. P. Süss. (*Pharm. Centr.*, xlvi., 159; through *Pharm. Journ.*, 1905, lxxiv., 589.)—Pimento berries which have been collected when nearly ripe become almost black in colour on drying, are deficient in aroma, and are covered with a saccharine exudation. Such unsightly berries are often artificially coated by means of a ferruginous substance, probably a clay or brown ochre. This may be detected by boiling the fruits with hydrochloric acid for half a minute, filtering, and testing the filtrate with potassium ferrocyanide solution. If the fruits be genuine only a bluish-green coloration is obtained, whilst a precipitate of Prussian blue is given in the case of artificially-coated berries. W. P. S.

The Composition of the Tubers of Edible Labiatæ. Balland. (*Journ. Pharm. Chim.*, 1905, xxi., 491-496.)—Numerous species of *Coleus* are cultivated in different parts of tropical Africa, and the tubers of two species of *Plectranthus* are also used for food. The specimens of tubers examined by the author resembled the potato in composition. They contained no sugar, and the ash was free from manganese.

Tubers of—	Water. Per Cent.	Nitrogenous Substances. Per Cent.	Fat. Per Cent.	Starch. Per Cent.	Cellulose. Per Cent.	Ash. Per Cent.	Phosphoric Acid. Per Cent.
Coleus Dazo ...	77·30	1·72	0·54	18·29	1·34	0·81	0·134
C. langouassiensis ...	87·10	1·59	0·09	10·07	0·52	0·63	0·134
C. rotundifolius (var. *alba*) ...	76·40	2·08	0·33	19·45	0·83	0·91	0·165
C. rotundifolius (var. *nigra*) ...	72·90	1·46	0·30	23·40	0·87	1·07	0·133
C. rotundifolius (var. *rubra*) ...	78·26	1·31	0·20	18·57	0·85	0·89	0·157
Plectranthus ternatus	71·30	2·74	0·39	22·69	1·26	1·62	—
P. tuberosus ...	77·00	1·52	0·26	18·90	1·05	1·27	—

C. A. M.

Characteristics of Apricot-tree Gum. P. Lemeland. (*Journ. Pharm. Chim.*, 1905, xxi., 443-448.)—Two samples examined by the author contained (1) 16·15 per cent. and (2) 16·50 per cent. of moisture, and yielded on ignition (1) 2·85 per cent. and (2) 3·4 per cent. of ash. One of the samples dissolved in water to the extent of 76·61 per cent., and the soluble portion had a specific rotation of $[\alpha]_D = -1{\cdot}93^\circ$. Oxydases were identified in both. The organic matter consisted chiefly of galactans and arabans, one sample yielding 19·8 per cent. of galactose and 38·87 per cent. of arabinose calculated on the original substance. C. A. M.

Colour Reactions produced by Vanillin with Hydrochloric Acid. L. Rosenthaler. (*Zeit. anal. Chem.*, 1905, xliv., 292-301.)—A tabular summary is given of different colorations recorded by previous observers as produced by this reagent. The reaction with acetone is particularly sensitive, and will detect 0·01 per cent. of that substance in the distillate from urine. The test is applied by mixing the 1 per cent. solution of vanillin in hydrochloric acid with an equal volume of sulphuric acid, adding one or two drops of the acetone solution, and heating the whole for fifteen minutes on the water-bath. The violet coloration can be easily distinguished from the red colour produced by the reagent alone under the same conditions. Lævulose and cane-sugar give a red coloration, whilst dextrose and lactose produce only a brown coloration. Most of the aromatic ketones tried gave negative results, and no coloration was obtained in cases in which a C_6H_5 group was in immediate combination with a CO group. Thus benzal acetone ($C_6H_5.CH=CH.CO.CH_3$) gave a coloration, but not benzalacetophenone ($C_6H_5.CH=CH.CO.C_6H_5$).

Some of the most frequently adulterated essential oils, such as clove oil, cinnamon oil, and aniseed oil, give little or no coloration with a 1 per cent. solution of vanillin in hydrochloric acid, whereas marked reactions are given by some of their adulterants, such as turpentine oil, and copaiba and gurjun balsams, and the addition of 10 per cent., and sometimes less, of these substances can be detected in this way. The reaction can also be used in testing Peru balsam, which gives no reaction either in the cold or on heating, whilst copaiba balsam gives a faint violet colour which gradually fades, and gurjun balsam gives an immediate purple-red colour, changing to violet. In each case the colour after heating is violet. It was found possible to detect 5 per cent. of gurjun balsam and 10 per cent of copaiba balsam in Peru balsam, and also to detect the presence of 10 per cent. of gurjun balsam in copaiba balsam, the faint coloration produced by the latter in the cold disappearing within fifteen minutes.

A comparison of the results led to the following conclusions: (1) Essential oils containing pinene or limonene ($C_{10}H_{16}$) give a green coloration with the reagent on heating, as is also the case with turpentine oil and other conifer oils. Sesquiterpenes such as cedrene, caryophyllene, and cadinene do not give the green coloration. (2) Oils containing linalool or geraniol or their esters give a violet coloration on heating. (3) The rose and violet colorations given by certain oils in the cold must be attributed to the presence of ketones or phenols. (4) Eucalyptol or Cineol give a blue coloration on heating, especially after the cooled liquid has been extracted with ether. In the case of oils containing eucalyptol the colour may be masked by the green colour due to terpenes.

Morphine and codeine give a reddish-violet colour on heating, whilst if antipyrin be present the liquid becomes orange, and green in the presence of picrotoxin, but in the latter case only after boiling for three or four minutes. C. A. M.

The Assay of Opium and its Preparations. W. H. Lenton. (*Pharm. Journ.*, 1905, lxxiv., 652, 653.)—In the process described the precipitated morphine is filtered off and titrated without previous weighing. For the assay of tincture of opium the details are as follows: The British Pharmacopœia process is followed up to the filtration of the alkaline liquid. Fifty c.c. are transferred by means of a pipette to a separating funnel, having a small plug of cotton-wool placed in the constriction above the tap. It is also well to deposit a thin layer of disintegrated filter-paper or asbestos fibre on the cotton-wool, and moisten the whole plug with morphinated water immediately before use. The usual quantities of ether, alcohol, and ammonium chloride are now added and the whole well shaken, care being taken not to dislodge the plug. After standing for the specified time the liquid is forced through the plug, air-pressure from an ordinary bellows being applied for this purpose. Ten c.c. each of ether and morphinated water are then added, shaken gently, and forced through the plug. This is repeated once again, after which the contents of the separator are washed with morphinated water until free from chloride, and finally with 2 c.c. of water. Twenty-five c.c. (or any convenient known quantity) of $\frac{N}{10}$ acid are now introduced into the separator, which is shaken thoroughly to dislodge the plug. The excess of acid is titrated back with $\frac{N}{10}$ sodium hydroxide solution, using methyl orange as indicator, as suggested by Dowzard (see ANALYST, 1904, xxix., 91).

W. P. S.

A New Reaction for Aconitine. E. P. Alvarez. (*Chem. News*, 1905, xci., 179-181.)—The following colour reaction, formed by the successive action of bromine, nitric acid, and potassium hydroxide on the alkaloid, is described. From 0·2 to 0·5 mgm. of the aconitine is treated in a small porcelain crucible with 5 drops of pure bromine, slightly heating the mixture on a brine-bath. One c.c. of fuming nitric acid is then added and the whole evaporated to dryness, a little more bromine being added when the acid loses its colour. About 0·5 c.c. of a saturated alcoholic solution of potassium hydroxide is now added to the yellow residue and again evaporated to dryness, when a red- or brown-coloured mass is obtained, according to the quantity of aconitine present. After allowing the crucible and its contents to cool, 5 or 6 drops of a 10 per cent. solution of copper sulphate may be poured over the residue. On moving the copper solution about it assumes a deep green colour. W. P. S.

The Detection of Antipyrin in Dimethylamido-antipyrin. P. Bourcet. (*Bull. Soc. Chim.*, 1905, xxxiii., 572-573.)—Dimethylamido-antipyrin, known commercially as pyramidon or amidopyrin, has ten times the selling value of antipyrin, and is, therefore, frequently adulterated with the latter, sometimes to the extent of a third of its weight. To detect this from 0·01 to 0·02 gram of the sample is dissolved in 4 to 5 c.c. of cold water, and the solution treated with 2 drops of sulphuric acid (66° Bé., S.G. 1·84) and 2 drops of a saturated solution of sodium nitrate or a pinch of the crystalline salt. On shaking the tube an immediate violet-blue coloration is produced, which rapidly disappears, leaving a colourless liquid when the dimethylamido-antipyrin is pure. If, on the other hand, antipyrin is present, the violet coloration gradually disappears, especially after the addition of more sodium nitrate, and is replaced by a pronounced bluish-green stable coloration, the intensity of which increases with the proportion of antipyrin in the mixture. In this way it is easy to detect 2 per cent. of antipyrin, and the nitroso compound formed is so slightly soluble in water that an approximate gravimetric determination can be made by modifying the conditions of the test. C. A. M.

Valuation of Aloes. A. Tschirch and R. Hoffbauer. (*Schweiz. Wochenschr. Chem. Pharm.*, 1905, xliii., 153; through *Chem. Zeit. Rep.*, 1905, xxix., 106.)—Several colour tests, both positive and negative, by which a good Cape aloe can be recognised are described. The active constituent may be quantitatively estimated in the following way: Five grams of the sample are digested in a small flask with 5 c.c. of methyl alcohol for two hours. The liquid is then warmed to 50° or 60° C., 30 c.c. of chloroform cautiously added with continued shaking, and the mixture allowed to stand for half an hour. The solution is filtered off into an Erlenmeyer flask, and the chloroform distilled. This extraction with chloroform is repeated four times. The total yellow residue finally obtained is dried at 100° C., and should not weigh less than 4 grams. A. N. C.

Detection of Potassium Hydrogen Tartrate in Potassium Antimony Tartrate. Schwartz. (*Repertoire*, 1905, xvii., 127; through *Pharm. Journ.*, 1905, lxxiv., 721.)—The presence of uncombined potassium hydrogen tartrate in tartar emetic may be detected by its action on sodium thiosulphate. A solution of the latter is decomposed by potassium hydrogen tartrate, sulphur being set free, whilst tartar emetic has no action. No turbidity should, therefore, be formed when a saturated solution of tartar emetic is mixed with an equal volume of $\frac{N}{10}$ thiosulphate solution and the mixture allowed to stand for five minutes. W. P. S.

ORGANIC ANALYSIS.

A Study of Methods for the Determination of Formaldehyde. R. H. Williams. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 596-601.)—The following conclusions, resulting from a critical examination of certain methods, practically confirm those previously arrived at by B. H. Smith (ANALYST, 1904, xxix., 5). The iodometric method is rapid and accurate, and is to be preferred for pure dilute solutions of formaldehyde. For concentrated impure solutions the peroxide method is the most satisfactory, but the time necessary for complete oxidation varies considerably, depending upon the concentration and temperature. The potassium cyanide method, which yields lower results than the oxidation methods, is to be recommended for dilute impure solutions. The end point of Legler's method is not satisfactory, which fact, as well as the low results, must be attributed to causes other than the influence of strong acids on hexamethylenetetramine. The discrepancy in the results yielded by the two different types of methods is due to conditions inherent in the methods themselves, and not to the presence of impurities. Apparently the condensation reactions are not complete, or a small part of the formic acid produced in the oxidation methods is further oxidized, giving high results. Paraformaldehyde when present behaves as formaldehyde. W. P. S.

A Colorimetric Method for the Detection and Determination of Formaldehyde. F. Bonnet, Junr. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 601-605.)—The method is based on the coloration obtained by the action of formaldehyde on morphine dissolved in sulphuric acid, first pointed out by Grünhut (*Zeit. anal. Chem.*, 1900, 329). To detect the presence of formaldehyde in milk, 60 c.c. of the latter are placed in a porcelain basin, and 1 c.c. of a solution containing 0·35 gram of morphine sulphate in 100 c.c. of concentrated sulphuric acid (specific gravity 1·84) is floated on the surface. The dish is then immediately covered with a glass plate. In a few minutes a coloration, varying from pink to blue, appears in the morphine solution should formaldehyde be present. In the case of minute traces (1 : 4,000,000) from one to two hours may elapse before the solution becomes slightly coloured. The test is applicable to curdled or sour milks, and also to butter, the latter being flattened over the bottom of the dish before adding the reagent.

By comparing the coloration obtained with those yielded by the same volume of milks containing known quantities of formaldehyde the test may be made quantitative. After ascertaining the approximate amount of formaldehyde present in the milk under examination, the determination is repeated, using as nearly as possible the correct quantity of formaldehyde in the control as is considered to be present in the sample, care being taken that the tests are started at the same time and made under like conditions. Over a range extending from 8 : 1,000 to 8 : 1,000,000, solutions containing the same amounts of formaldehyde give the same characteristic coloration in the same period of time. The other ordinary milk preservatives give no coloration with this reagent. W. P. S.

On the Volatility of Lactic Acid in Steam. F. Utz. (*Chem. Zeit.*, 1905, xxvii., 363.)—Many authorities hold that there is no appreciable loss of lactic acid on boiling an aqueous solution, but in some experiments on milk the author has been led to disagree with this opinion.

The results of his experiments show that lactic acid is volatile in steam, and to a greater degree in concentrated than in dilute solutions, but that it is not so volatile that the lactic acid can be completely distilled from an aqueous solution, though, according to A. Partheil and others, this is possible in a concentrated solution by the use of superheated steam (cf. Bevan, ANALYST, vol. xix., 242). A. N. C.

Detection of Palm Oil when used as a Colouring Material in Fats and Oils. C. A. Crampton and F. D. Simons. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 270-274.)—The high natural colour of palm oil has led to its use as a means of imparting a butter-like colour to oleomargarine. Small quantities of this oil may be detected in margarine and butter by the following tests: 100 c.c. of the fat are dissolved in 300 c.c. of light petroleum, and shaken out with 50 c.c. of 0·5 per cent. potassium hydroxide solution. The aqueous layer is separated, acidified, and shaken out with 10 c.c. of carbon tetrachloride. The carbon tetrachloride solution is treated with 2 c.c. of a solution of 1 part of crystallized phenol in 2 parts of carbon disulphide, and 5 drops of hydrobromic acid (specific gravity 1·19). The almost immediate development of a bluish-green colour is indicative of palm oil. Ten c.c. of the melted and filtered fat when shaken with 10 c.c. of acetic anhydride and 1 drop of sulphuric acid (specific gravity 1·53) impart a blue coloration to the lower layers of the mixture on settling if the fat contain palm oil. Sesamé oil gives a similar coloration, but not after previous extraction with alcohol. Extraction of palm oil with alcohol has no effect on the formation of the colour.

In each test the coloration is transient; changes which occur after the lapse of a few minutes are therefore to be disregarded. The above reactions will detect the presence of about 0·5 per cent. of palm oil in other oils or fats. W. P. S.

The Characteristics of Stearin Pitches. E. Donatti. (*Chem. Rev. Fett-u. Harz-Ind.*, 1905, xii., 42-44, 73-75.)—The commercial products sold as "stearin pitch" are of various origin. Those obtained from the distillation of crude fatty acids in the candle industry are viscid black pitches, which when distilled yield fluorescent inflammable oils. Other "stearin pitches," prepared from the fatty acids separated from wool-washings are brownish or black residues, differing considerably in characteristics from true stearin pitch and from wool pitch, and the author therefore proposes to term them "stearin wool pitch." Wool pitch and stearin wool pitch are characterised by containing dark neutral compounds resembling asphaltum. These are partially soluble in ether, and dark cohesive precipitates are formed on adding alcohol to the solution. Stearin wool pitch yields much more ash than fat pitch, and the presence of calcium sulphate in large proportion indicates its preparation from wool washings. The undecomposed fatty acids, etc., can be extracted in a Soxhlet's apparatus by means of ether or petroleum spirit, and the solution on evaporation leaves a brownish fat-like residue. Further extraction with benzene yields dark solutions, from which on evaporation brilliant black solid or viscid residues are obtained, very similar to those yielded by asphaltum. Only a small residue is left after the two extractions, and this dissolves in carbon bisulphide, with the exception of the mineral matter, in which respect it resembles that from some natural asphaltums and other pitches.

As regards the valuation of these pitches, the melting-point and proportion of neutral fats and fatty acids are important criteria, whilst the proportion of constituents of an asphaltum nature is of primary importance when the pitch is required for the manufacture of varnishes for cables and insulating materials. Ash or decomposition products insoluble in ether or benzene can never be regarded as an advantage. Two samples of stearin wool pitch examined by the author had acid values of 14·07 and 16·07, and saponification values of 46·56 and 70·32 respectively. On treatment with ether they yielded 52·84 and 77·56 per cent. of insoluble matter respectively, whereas true stearin pitches yielded from 0·97 to 8·67 per cent. C. A. M.

Determination of the Melting-points of Pitch, Asphalt, etc. M. Wendriner. (*Zeits. f. angew. Chem.*, 1905, xviii., 622.)—The method is a modification of one due to Kraemer and Sarnow, in which the melting-point of the pitch was taken to be the temperature at which mercury broke through a stopper of the pitch in a tube heated in a water-bath. The author found, however, that the process gave varying results for the same sample of pitch; he therefore suggests the modification that, in order to avoid differences in the rate of heating, the pitch should be put into a bath heated a few degrees above the melting-point, and not into a bath which is gradually heated up after the tube of pitch has been placed in. The use of an air-jacket is then desirable.

The author gives figures to show that the melting-point of a sample of pitch can be consistently and accurately determined by his process, and he also gives some figures showing the effect of varying the size of the pitch-stopper, the quantity of mercury, and the temperature of the water-bath.

The author describes the apparatus he uses, which can be obtained from C. Gerhardt, Bonn. For details the reader is referred to the original paper.

E. K. H.

The Detection of Sodium Silicate in Soap. Ahmed Hussein. (*Journ. Pharm. Chim.*, 1905, xxi., 496, 497.)—In the ordinary method the soap is dissolved in hot alcohol, the insoluble residue dried in the oven, dissolved in water, and the solution treated with hydrochloric acid, which should give a precipitate of silica. The author, however, finds that sodium silicate after being separated by means of alcohol and dried becomes almost insoluble in water, and so may escape detection. He therefore recommends heating the residue with a little water to which has been added a trace of sodium hydroxide, to bring the silicate into solution. The liquid is then filtered, and hydrochloric acid added until the reaction is acid, the silica still remaining in solution. Ammonia is now added until the liquid is alkaline and the silica is precipitated. C. A. M.

INORGANIC ANALYSIS.

Estimation of the Halogen in Halogen Salts of Mercury. T. Fischer. (*Chem. Zeit.*, 1905, xxvii., 361.)—The ordinary method of estimating the bromine by precipitation with silver nitrate after the removal of the mercury with sulphuretted hydrogen was found in some analyses of double salts to be both troublesome and inaccurate. After some unsuccessful experiments, a distillation method was worked out, which gives good results, for either bromine or chlorine.

To the mercury salt in a distilling flask 5 c.c. of 10 per cent. soda are added, and warmed for twenty minutes on the water-bath. After cooling, 3 c.c. of (1 : 1) sulphuric acid are added, and after another cooling 0·4 gram of potassium permanganate suspended in 10 c.c. (1 : 1) sulphuric acid. The flask is quickly connected to a receiver containing a solution of potassium iodide, and boiled for five minutes, when all the bromine will have been distilled over. The solution in the receiver is acidified with two drops of sulphuric acid, and the freed iodine titrated in the usual way with $\frac{N}{20}$ sodium thiosulphate.

For iodine, another method must of course be used, the separation of the mercury being accomplished by the use of magnesium powder.

The iodine salt is mixed in a small flask with magnesium powder and 10 to 20 c.c. of water. On shaking this mixture, magnesium iodide and magnesium amalgam are formed, the latter being quickly converted into magnesium hydroxide and mercury with evolution of hydrogen by the water present. The reaction lasts for one or two minutes, and the solution is then filtered, acidified with chlorine free nitric acid, and the iodine precipitated as silver iodide. This is reduced to silver in a current of gas, with the usual precautions, and weighed. A. N. C.

The Determination of Acetic Acid in White Lead. G. W. Thompson.

(*Journ. Chem. Ind.*, 1905, p. 487.)—Having found the published methods for this estimation unsatisfactory, the author has worked out a direct distillation method, which gives him good results.

Eighteen grams of the dry white lead are mixed with 40 c.c. of syrupy phosphoric acid, 18 grams of zinc-dust, and 50 c.c. of water in a 500 c.c. distilling-flask connected with a steam supply.

The liquid is alternately distilled down to a small bulk, and made up again to about 200 c.c. by passing in steam till 10 c.c. of the distillate require but 1 drop of $\frac{N}{10}$ alkali to affect phenolphthalein. The total distillate is then titrated against $\frac{N}{10}$ alkali.

A. N. C.

The Determination of Copper and Free Matte in Scoriæ. Vallety.

(*Ann. de Chim. anal.*, 1905, x., 193.)—The method is based upon the behaviour of silver nitrate towards the different products: (1) Copper displaces silver completely from its solutions. (2) Oxide of copper on treatment with silver nitrate gives a precipitate of basic nitrate which dissolves in 5 per cent. nitric acid. (3) Silver nitrate reacts with the matte (ignited Cu_2S), yielding silver, silver sulphide, and copper nitrate. (4) Silver nitrate does not react to any appreciable extent with copper silicate. In the analysis 5 grams of the scoriæ (enclosing 0·75 per cent. of copper) are treated with 100 c.c. of a solution of silver nitrate (50 grams per litre), and 5 c.c. of nitric acid subsequently added. After twelve hours' digestion the liquid is filtered, the residue washed with water containing 3 per cent. of nitric acid, and the copper in the filtrate and washings determined by the usual methods. C. A. M.

Estimation of Arsenic in Fuels. G. McGowan and R. B. Floris.

(*Journ. Chem. Ind.*, 1905, xxiv., 265.)—When a precipitate of arsenious sulphide is boiled with water, the sulphide is converted quantitatively into oxide, which remains in solution.

This process is used to shorten the authors' method for the estimation of arsenic described in the final Report of the Royal Commission. In the method as modified the powdered fuel is ignited with its own weight of lime for three or four hours. The residue is digested with a little nitric acid, dried, and again ignited. This residue is dissolved in dilute hydrochloric acid, the solution filtered, the filtrate reduced with aqueous sulphurous acid, boiled, saturated with sulphuretted hydrogen, and allowed to stand for several days. The precipitate is then filtered in a Gooch crucible, and boiled with its asbestos for three hours in 200 c.c. of water.

A fourth part of this extract is "marshed," and the mirror obtained compared with a standard. The results compare well with those obtained by the original method.

A. N. C.

On the Estimation of Sulphur in Burnt Pyrites. W. Jene.

(*Chem. Zeit.*, 1905, xxvii., 362.)—In many works-laboratories the sulphur is still estimated by digesting the sample with nitric acid, the excess of nitric acid removed by repeated evaporation with hydrochloric acid, and the sulphuric acid in the filtered solution directly precipitated with barium chloride.

Analyses made by the author show that the results by this method are lower than those obtained by the fusion method of Fresenius, sometimes by as much as 40 per cent., and that this difference is due, not to a loss of sulphur as sulphuretted hydrogen, but to the incomplete nature of the acid extraction. The author recommends fusion of the pyrites with sodium peroxide, a method giving excellent results.

A. N. C.

On the Determination of Zirconium in Presence of Titanium, especially in Rocks. M. Dittrich and R. Pohl. (*Zeits. anorg. Chem.*, 1905, xliii., 236.)—The authors' method is based on a direct weighing of the mixed oxides of zirconium and titanium, followed by a colorimetric determination of titanium, zirconium being then found by difference. In the analysis of rocks, the ignited ammonia precipitate obtained as usual is fused with caustic soda to remove alumina and phosphoric acid. The residue left on dissolving the melt in water is fused with potassium bisulphate and dissolved in cold water, iron and manganese being then precipitated as sulphides by ammonium sulphide in the presence of tartaric acid. The filtrate from these sulphides, which contains only titanium and zirconium, is evaporated to dryness, the residue taken up in dilute sulphuric acid, and the tartaric acid destroyed by the cautious addition of potassium persulphate, followed by heating. The liquid is then evaporated until nearly all the sulphuric acid has been volatilized, the residue is dissolved in hydrochloric acid and water, and titanium and zirconium hydroxides precipitated by ammonia. The precipitate is washed, re-dissolved, re-precipitated, ignited, and weighed as $TiO_2 + ZrO_2$. The oxides are then fused with potassium bisulphate, and titanium dioxide determined colorimetrically according to Weller (*Ber.*, 1882, xv., 2592).

No test analyses of rocks are given. Results obtained on pure solutions containing iron, titanium, and zirconium show a maximum error of about 3 mgms. on the oxides, the quantities used being about 0·1 gram of Fe_3O_4, and 0·01 gram of each of the other two oxides. Better results are obtained if both the iron and the mixed titanium and zirconium oxides are finally purified from silica by a potassium bisulphate fusion. In this case the error on each oxide only amounts to a few tenths of a mgm.

A. G. L.

A Reaction of the Compounds of Rhodium of Use in Chemical Analysis. E. Alvarez. (*Chem. News*, 1905, 216.)—The author describes a reaction by which the formation of the blue sodium per-rhodate (RhO_4Na_2) serves easily to distinguish this metal from the others of the same group.

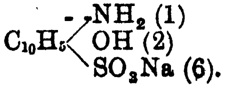
To an aqueous solution of any soluble rhodate an excess of soda is added, and the liquid exposed to the gaseous mixture produced by the action of cold concentrated hydrochloric acid on potassium chlorate.

The dilute and almost colourless solution turns through yellow to red, and as the current of gas continues a faint green precipitate appears, dissolving again to a bright blue solution. This is the per-rhodate of sodium of Claus.

Peroxide and persulphate of sodium decolorize the liquid with evolution of oxygen.

A. N. C.

A New Reagent for Potassium. Eugenio Pinerúa Alvarez. (*Chem. News*, 1905, xci., 146.)—The reagent described consists of a 5 per cent. solution of "iconogene," sodium amidonaphthol sulphonate,



$$C_{10}H_5\left\langle\begin{array}{l}NH_2\ (1)\\ OH\ (2)\\ SO_3Na\ (6).\end{array}\right.$$

The solution should be freshly prepared with cold boiled-out distilled water just before using. With all potassium compounds it gives in neutral solutions a precipitate of the white, very brilliant, crystalline potassium amidonaphthol sulphonate, which is slightly soluble in water and insoluble in alcohol. The crystals consist of orthorhombic plates. The reaction requires some little time, and if less than 2 per cent. of the potassium salt is present, the crystals will not form for several hours. The delicacy of the test is stated to be as great as that of the platinum chloride method. An advantage of iconogene is that no precipitate is obtained with ammonium salts; magnesium also does not interfere if sufficient ammonium chloride is present. Iron and manganese give no precipitate with iconogene; nickel, cobalt, and bismuth give a precipitate insoluble in excess; copper gives a precipitate soluble in excess of the reagent with a green colour. A. G. L.

The Solubility of Barium Sulphate in Chromic Chloride Solution. F. W. Küster and Georg Dahmer. (*Zeits. anorg. Chem.*, 1905, xliii., 348.)—The authors found, on boiling 1·4198 grams pure barium sulphate with 15 c.c. of a fairly concentrated solution of chromic chloride, that 0·0015 gram passed into solution. On treating 4·4063 grams barium sulphate in the same way, 0·0051 gram was dissolved. On boiling 15 c.c. of chromic chloride solution acidulated with hydrochloric acid with 1·0262 grams barium sulphate, 0·0176 gram of the latter was dissolved after ten days. A. G. L.

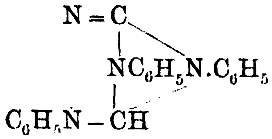
The Estimation of Potash in Soils, Plants, and Fertilizers. F. P. Veitch. (*Journ. Amer. Chem. Soc.*, xxvii., 56.)—The author has examined Moore's method (*Journ. Amer. Chem. Soc.*, xx., 342) for determining potash in soils, plants, and fertilizers. The method consists in dissolving the sample in acid, destroying ammonium salts and organic matter by means of aqua regia, and evaporating repeatedly with hydrochloric acid. The residue is extracted with water and a few drops of hydrochloric acid, a slight excess of platinic chloride is added, and the whole evaporated nearly to dryness on a water-bath. The evaporation must not be carried to complete dryness, which would render the iron and aluminium chlorides difficultly soluble. The cold mass is treated with 15 c.c. to 25 c.c. of acidified 90 per cent. alcohol, stirred and filtered after a few minutes, being washed with alcohol and ammonium chloride solution. The potassium platinochloride obtained is dried, dissolved in hot water, and weighed as usual. The alcohol used is prepared by passing dry hydrogen chloride into 90 per cent. alcohol until 1 c.c. of the alcohol neutralizes about 2·3 c.c. of normal alkali solution.

For soils, the author found that the results given by the method as described above, and by a modification in which ordinary 90 per cent. alcohol was substituted for the acidified alcohol, were practically identical with those given by the official method. For fertilizers, on the other hand, the method always gave somewhat higher results (maximum difference, 6·19 per cent. K_2O, instead of 5·70), and it was found that whilst more concordant results could be obtained by destroying the organic matter by ignition in a porcelain dish instead of by aqua regia, yet in this case the residue obtained required digestion with hydrochloric acid, occasionally for some time, in order to extract all the potash. The author recommends the Moore method, original or modified, for soils, but sees no advantage over the official method in using it for fertilizers. A. G. L.

The Detection of Sodium Salts by Frémy's Reagent. J. Bougault. (*Journ. Pharm. Chim.*, 1905, xxi., 437-442.)—The following modification of Frémy's reagent is easily prepared, and is stated to give constant results: A mixture of 10 c.c. of pure potassium hydroxide solution (33·3 per cent.) and of 45 c.c. of hydrogen peroxide (10 vol. solution) is gently heated with 1 gram of antimonous chloride until, after five to ten minutes, the solution is complete, with the exception of a very slight amorphous sediment, from which the liquid can be decanted. In using the reagent the following precautions should be observed: (1) A very small amount (0·5 c.c.) should be used. (2) The solution of the sodium salt should be as concentrated as possible, and should be neutral or alkaline. (3) Boiling the mixed solutions for a few seconds facilitates the formation of the precipitate. The reagent gives an immediate precipitate with 0·4 mgm. of sodium chloride. Potassium salts interfere with the sensitiveness of the reaction, and should be eliminated as far as possible, preferably by treatment with tartaric acid. Lithium salts react like sodium salts, and 0·05 gram of lithium nitrate in 1 c.c. of water gives an immediate precipitate of lithium pyroantimonate with 1 c.c. of the reagent. This crystallizes in microscopic hexagonal lamellæ; whereas the sodium compound, if deposited slowly, forms prisms, approximating to cubes, or octahedra, or, if deposited rapidly, is in the form of needles united together at the top, or of small elongated prisms.

C. A. M.

A Gravimetric Method of Determining Nitric Acid. M. Busch. (*Berichte*, 1905, xxxviii., 861-866.)—This is based upon the fact that diphenyl-endanilo-dihydrotriazole—



which is sold by Merck under the name of *nitron*, forms an insoluble salt with one molecule of nitric acid, a crystalline precipitate being formed on treating 5 to 6 c.c. of the nitrate solution with a few drops of a 10 per cent. solution of the reagent in dilute acetic acid (5 per cent.). Insoluble compounds are also given by hydrobromic, hydriodic, nitrous, chloric, perchloric, chromic, and thiocyanic acids, so that the absence of these must be insured before testing for nitric acid. The reagent is capable of detecting 1 part of nitric acid in 60,000. For a quantitative determination the substance, which should contain about 0·01 gram of nitric acid, is dissolved in 80 to 100 c.c. of water, and the solution, after the addition of a few drops of dilute sulphuric acid, is heated to the boiling-point and treated with 10 to 12 c.c. of the acetic acid solution of the reagent. The flask is then kept for one and a half to two hours in ice water, after which the contents are filtered with the aid of a pump, and the precipitate washed with 10 to 12 c.c. of iced water, and dried for forty-five minutes at 110° C. The weight of the precipitate multiplied by 0·168 gives the weight of the HNO_3. When nitrous acid is present with nitric acid it may be destroyed by a preliminary treatment with hydrazine sulphate, but the results for nitric acid subsequently obtained by the above method will then be slightly too high. C. A. M.

The Determination of Nitrogen as Nitrites in Waters. R. S. Weston. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 281-287.)—The following modification of the naphthylamine method is recommended: The two solutions required are prepared by dissolving 8 grams of sulphanilic acid in 1 litre of dilute acetic acid (specific gravity 1·044), and 8 grams of α-naphthylamine also in 1 litre of this dilute acid. It may be necessary to warm the latter solution to dissolve the salt, and afterwards to filter it. Two c.c. of each of these reagents will be found sufficient for 100 c.c. of the water. The coloration produced is compared with that given by known quantities of standard nitrite solution. Excess of acetic acid does not interfere with the delicacy of the reaction, and is to be preferred to hydrochloric acid in making up the reagent. The water and reagents should be stirred after mixing to prevent the dense acid solution falling to the bottom of the test cylinder. W. P. S.

Colorimetric Determination of Phosphorus. Thomas E. Hewitt. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 121.)—The method depends on the colour given with hydrogen sulphide by alkaline solutions of ammonium phosphomolybdate. To carry it out, the phosphomolybdate precipitate is obtained as usual from 2 grams of the steel or iron. The precipitate is washed with 2 per cent. nitric acid. The filter and precipitate are then placed over a 100 c.c. measuring-flask, and the precipitate is treated first with hot water and then with sodium hydroxide solution (4 grams NaOH per litre) until just dissolved. Half as much again of the sodium hydroxide solution is then added, and the flask is filled to the mark with water. An aliquot part of the solution is placed in a 50 c.c. Nessler tube, diluted to 25 c.c., and hydrogen sulphide is passed through the liquid for five minutes. After allowing the tube to stand in boiling water for another five minutes, it is filled to the mark with water, and its colour compared with a similar tube containing 10 c.c. of standard phosphomolybdate solution similarly treated. The colours should be compared by looking downwards into the tubes, light being reflected upwards through them. The treatment with boiling water renders the colours permanent for about two hours. The standard phosphomolybdate solution is made by dissolving 0·2737 gram of dry ammonium phosphomolybdate in a 50 per cent. excess of the sodium hydrate solution, and diluting to 500 c.c. with water. Of this solution, 10 c.c. = 0·000009122 gram phosphorus. Several determinations by this method on each of four samples of iron and steel gave results in most cases within 0·001 per cent. of those obtained by titration with standard sodium hydrate. In the case of a Bessemer pig-iron, however, containing 0·102 per cent. of phosphorus, the results varied between 0·089 and 0·083 per cent.; and another similar sample, containing 0·088 per cent. of phosphorus, showed 0·083 to 0·079 by this method. A. G. L.

Methods of Titration of Phosphoric Acid. O. J. Hlavnicka. (*Zeits. f. angew. Chem.*, 1905, xviii., 655.)—The author claims that Hundeshagen's method, as compared with other volumetric processes for the estimation of phosphoric acid, is simple and practical. The method, in detail, used in the author's laboratory is as follows: The phosphate is precipitated as $MgNH_4PO_4$ by the citrate method, and the precipitate is collected with the aid of suction on a hardened filter-paper, washed first with $2\frac{1}{2}$ per cent. ammonia, and then with alcohol till free from ammonia. The total volume of the washings need not be more than about 40 c.c. The precipitate is then washed off with cold water into a porcelain dish (total volume required about 200 c.c.), and, after the addition of a few drops of methyl orange solution (strength, 0·1 per cent.), decomposed with a slight excess of $\frac{3}{10}$-N HCl, and the excess found by titration with $\frac{3}{10}$-N NaOH (1 c.c. $\frac{3}{10}$-N = 0·01065 gram P_2O_5).

For this process the amount of substance taken should be such as to yield about 0·15 – 0·2 gram P_2O_5. The estimation in the case of superphosphate takes from two to two and a half hours. This method, the author finds, gives results agreeing with those obtained by the gravimetric molybdate method, but about 0·1 to 0·2 per cent. higher than the gravimetric citrate method figures. This result was also obtained by Hundeshagen (*Chem. Zeit.*, xxv., 446).

Although the method due to Raschig (ANALYST, this vol., p. 221), where pure water is used for washing the $MgNH_4PO_4$ precipitate, is simpler, the author considers the above method better adapted for technical analyses, as a greater volume of washing is permissible, and the use of an ordinary hardened filter is also an advantage.

E. K. H.

The Determination of Free Phosphoric Acid in Superphosphates. Gerhardt. (*Chem. Zeit.*, 1905, xxix., 178.)—A process depending on the reaction of phosphoric acid with calcium carbonate, according to the equation—

$$2H_3PO_4 + CaCO_3 = H_2O + CaH_4(PO_4)_2 + CO_2,$$

and which consists in treating the superphosphate with excess of calcium carbonate, and estimating either the CO_2 evolved or the unused $CaCO_3$ remaining. The presence of small amounts of iron sulphate in the sample forms a source of error, as this substance also reacts with calcium carbonate, rendering the result low. This error can be avoided by precipitating the iron with potassium ferrocyanide.

The analytical process is as follows: 20 grams of the sample are shaken with water in a litre flask for half an hour. One gram of potassium ferrocyanide dissolved in water is added, and the solution made up to the litre. One hundred c.c. are filtered, and shaken with an excess of calcium carbonate for one half-hour, again filtered, washing the precipitate only slightly, and the filtrate evaporated to dryness. The residue, consisting of calcium phosphate, is carefully ignited and weighed. Or the process can be carried out in a Scheibler's or similar apparatus, and the resulting CO_2 estimated.

The author recommends, however, the volumetric estimation of the excess of carbonate. For this purpose the solution, after the reaction has been completed, is treated with 25 c.c. of $\frac{N}{1}$ hydrochloric acid, made up to 200 c.c., and 100 c.c. filtered through a dry paper. This is titrated with $\frac{N}{2}$ NaOH, using methyl orange as indicator. The colour change is sharp, and this method is certainly the most accurate one.

H. A. T.

Estimation of Combined Sulphuric Acid by the Methods of Lunge and of Silberberger. (*Report of the Ninth Sub-Committee of the International Analysis*

Commission. Drawn up by G. Lunge. Zeits. f. angew. Chem., 1905, xviii., 449; *Berichte*, 1903, xxxvi., 2755.)—Silberberger criticised Lunge's method, and suggested a new method, using strontium chloride in place of the barium salt used by Lunge. This new method has been investigated and found unreliable, and therefore distinctly inferior to the original method devised by Lunge. E. K. H.

The Titration of Sulphurous Acid in Alkaline Solution. O. Ruff and W. Jeroch. (*Berichte*, 1905, xxxviii., 409-419.)—Direct titration of sulphurous acid by means of iodine solution in the presence of sodium bicarbonate yields incorrect results, owing to the oxidation of the sulphite through the agency of the iodine ions which act catalytically. On the other hand, the method in which an excess of iodine is added, and the excess titrated with thiosulphate solution, is also shown to be unreliable, and the authors therefore recommend the addition of 10 to 20 per cent. of mannitol to the liquid, and carrying out the titration in an atmosphere of carbon dioxide to prevent oxidation. Direct titration is preferable, but if iodine be added in excess, arsenious acid should be used in place of thiosulphate for the back titration.

C. A. M.

On the Analysis of Sodium Hydrosulphite. A. Binz and H. Bertram. (*Zeits. angew. Chem.*, 1905, xviii., 168.)—The authors have compared Ekker's method (*Rec. trav. chim. Pays-Bas*, 1894, xiii., 36) for the determination of hydrosulphite with other methods, especially with the indigo method, and find it convenient and reliable. In a solution in which by the indigo method 2·938 grams $Na_2S_2O_4$ per litre were found, Ekker's method gave values varying from 2·818 to 2·970. The method is carried out by dissolving the hydrosulphite in air-free water in an atmosphere of carbon dioxide, adding a drop of ferrous sulphate solution, and running in standard potassium ferricyanide solution until a blue colour appears, air being carefully excluded all the time. The oxidation proceeds according to the equation:

$$Na_2S_2O_4 + 2K_3Fe(CN)_6 = 2SO_2 + 2K_3NaFe(CN)_6.$$

Since Turnbull's blue is decomposed by alkalies, a little acetic acid should be added to the liquid, if so much alkali is present in the sample that the sulphur dioxide formed during the reaction is insufficient to neutralize it. A. G. L.

A New Method of separating Chlorides, Bromides, and Iodides in Mixtures. O. Wentzki. (*Zeits. f. angew. Chem.*, 1905, xviii., 696.)—This method depends upon the fact that when alkali iodides are shaken up with mercurous chloride or bromide, double decomposition takes place, and mercurous iodide is precipitated, whilst an equivalent quantity of chloride or bromide goes into solution; in the same way, alkali bromides, when shaken with mercurous chloride, yield mercurous bromide and an equivalent of a soluble chloride. These reactions are quantitative.

These facts have been applied to a separation of any combination of chlorides, bromides and iodides by the author. Examples of the four possible combinations are given, but the method of working and of calculation will be clear if the most complex case only is here described—that, namely, of a mixture of the three.

In an actual experiment, a solution containing per litre 0·585 gram $NaCl$, 1·19 gram KBr, and 1·66 gram KI was used, and the following determinations were made:

(1) One hundred c.c. were precipitated with silver nitrate; the weight

$$AgCl + AgBr + AgI = 0{\cdot}5640 \text{ gram.}$$

(2) Three hundred c.c. were shaken in a stoppered flask with mercurous bromide until in a filtered small sample iodine could not be found by nitrous acid and chloroform. One hundred c.c. of the filtrate were precipitated with silver nitrate; weight of $AgCl + AgBr = 0{\cdot}5175$ gram.

(3) Three hundred c.c. of the original solution were shaken with mercurous chloride, until neither iodine nor bromine could be detected in a filtered sample. One hundred c.c. of the filtrate were then precipitated with silver nitrate, and the weight $AgCl = 0{\cdot}4294$ gram.

As the iodide in (1) has been replaced in (2) by its equivalent of bromide, the difference between (1) and (2) × the ratio $AgI : (AgI - AgBr)$, *i.e.*, 5·007, gives the weight of AgI in the first precipitate = 0·2327.

Similarly, the difference between (2) and (3) × the ratio $AgBr : (AgBr - AgCl)$ *i.e.*, 4·221, gives the weight of $AgBr$ contained in the second precipitate = 0·3718. Then by subtraction of this value from the weight of (2), we get the weight of $AgCl$ contained in (2) = 0·1457. This quantity is also contained in the first precipitate, and we have already found the weight of AgI therein, hence:

$$\begin{array}{lr} AgCl + AgBr + AgI & = 0{\cdot}5640 \\ AgCl\ 0{\cdot}1457 + AgI\ 0{\cdot}2327 & = 0{\cdot}3784 \\ \hline AgBr & = 0{\cdot}1856 \end{array}$$

From these figures we get:

	Chlorine.	Bromine.	Iodine.
Found	0·0360	0·0790	0·1257
Calculated	0·0354	0·0799	0·1268

The author found the process hold equally for sodium, potassium, ammonium, magnesium and alkaline-earth salts.

E. K. H.

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REVIEW.

The Brewing Industry. By Julian L. Baker, F.I.C., F.C.S.

This little work is published by Methuen and Co., and constitutes one of their well-known series of "Books on Business." As such it has not been written for the expert, but is intended to convey to any member of the lay public who may consult its pages useful information in connection with the materials used in brewing, and a clear notion of the more important principles underlying the operations of the brewery. Mr. Baker's qualifications for the task he has undertaken are well recognised, and this little book, which is both accurate in matter and clear in arrangement, should do much to dissipate the ignorance and misconception which prevail so extensively in connection with the manufacture of our national beverage. The book contains very few errors, typographical or otherwise, and has a number of well-executed illustrations.

A. C. C.

Erratum.—Page 184, second line from top, *for* "50 c.c." *read* "5 c.c."

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NOTE ON THE SEPARATION OF STRYCHNINE AND BRUCINE.

By D. LLOYD HOWARD.

(*Read at the Meeting, June* 7, 1905.)

As a preliminary to the investigation of Gordin's method for the separation of strychnine and brucine, and the determination of the former in presence of the latter, it appeared desirable to investigate Keller's method, of which that of Gordin is a modification. It is the account of this preliminary investigation which I have the honour to submit to the Society. Keller's method is as follows :

"0·2 to 0·4 gram of the dry mixture of alkaloids is dissolved in 10 c.c. of 10 per cent. sulphuric acid by heating on the water-bath. After complete cooling, 1 c.c. of nitric acid S.G. 1·40 to 1·42 is added, and the mixture shaken. The strychnine sulphate, which usually separates from the solution, is redissolved on addition of the nitric acid, and at the same time the red brucine-nitric acid coloration is developed. After allowing the mixture to stand for an hour to an hour and a half, in order to complete the destruction of the brucine, 40 grams of chloroform, an equal quantity of ether, and 10 c.c. of 10 per cent. ammonia are added, and the whole shaken. Forty grams of the extract are filtered into a tared flask, from which the mixture of chloroform and ether are distilled. The residual strychnine is dried at 95° to 100° C. and weighed." It has been stated that this process "only returns about 96 per cent. of the alkaloid," but on experimenting with a mixture of strychnine and brucine the results were found to be too high—*e.g.*, 0·2486 gram strychnine with 0·19 gram brucine gave 0·2498 gram residue = 100·48 per cent. It should be mentioned that the acid solution was extracted three times with the solvent mixture, the latter washed with ammonia solution (which separates more easily than water), and the whole of the solvent distilled.

Brucine similarly treated left a small residue (probably due to some impurity in the alkaloid used, as it gave no reaction for brucine), but not enough to account for the error in the strychnine determination described above.

E.g., 0·412 gram brucine gave a residue of 0·002 gram = 0·5 per cent. A number of experiments were then made, using strychnine only.

Using caustic soda solution to precipitate the alkaloid and to wash the solvent, 0·2434 gram strychnine gave 0·2473 gram residue = 101·6 per cent.

A large excess of caustic soda was then tried—viz., 30 c.c. of 10 per cent. solution—with the result that 0·4617 gram strychnine gave 0·4629 gram residue = 100·23 per cent.

Discarding the nitric acid, and dissolving the strychnine in sulphuric acid, extracting, and washing the solvent with ammonia solution, 0·2776 gram strychnine gave 0·2795 gram residue = 100·7 per cent.

Washing the solvent twice with ammonia solution, S.G. 0·959, 0·3311 gram gave 0·3358 gram residue = 101·4 per cent.

On filtering the solvent after washing with ammonia the same result was obtained—viz., 101·4 per cent.

Drying the solvent with calcium chloride, 0·2909 gram strychnine gave 0·2948 gram residue = 101·3 per cent., but the residue contained a trace of chloride.

An experiment was made with strychnine recrystallized from alcohol and dried at 50° C. till it ceased to lose weight; 0·3068 gram gave 0·3097 gram residue = 100·9 per cent.

Using chloroform only as a solvent without treatment with acid, 0·3677 gram gave 0·3710 = 100·9 per cent. after drying for twelve hours at 100° C., and this was not reduced by further drying at 110° C.

A similar experiment, using ether (purified by washing with chromic acid, sodium bisulphite, and caustic soda, and then by distilling); 0·3802 gram gave 0·3831 gram residue = 100·7 per cent.

A similar experiment, using a mixture of ether and chloroform, 0·2364 gram gave 0·2384 gram residue = 100·8 per cent.

On the other hand, dissolving strychnine in alcohol, then distilling off the latter, produced no alteration in the weight; also redissolving the residue from the ether or chloroform extractions in alcohol, and distilling off has little or no effect on the result.

A mixture of benzene and amylic alcohol (which is an excellent solvent for cinchona alkaloids) was tried as a solvent, but proved unsatisfactory, the residue after distilling being a brown resinous body with strongly-marked odour, and the results being very high—*e.g.*, 0·3217 gram gave 0·3365 gram residue = 104·6 per cent.

As these results do not bear out the statement that Keller's method only returns about 96 per cent. of the alkaloid, some experiments were made to ascertain the effect of temperature on the reaction.

0·1612 gram strychnine with 0·2017 gram brucine, left in contact with the nitric acid for three and a half hours at 21° C., gave only 0·1456 gram residue, or 90·32 per cent.

0·2256 gram strychnine with 0·1785 gram brucine, left in contact with the nitric acid for seventeen hours at 21° C., gave only 0·1821 residue, or 80·7 per cent.

0·2412 gram strychnine with 0·1452 gram brucine, left in contact with the nitric acid for seventeen hours at 0° C., gave 0·2420 gram residue, or 100·3 per cent.

0·3436 gram strychnine with 0·1273 gram brucine, left in contact with the nitric acid for two hours at 0° C., gave 0·3460 gram residue = 100·7 per cent., almost exactly the result obtained by dissolving strychnine in the chloroform and ether mixture, and then distilling without any treatment with acid.

It appears, therefore, that at a sufficiently low temperature brucine can be completely destroyed in the presence of strychnine without injury to the latter, and that the defects of Keller's method are simply those attendant on the use of chloroform and ether as a solvent.

I feel that I owe some apology to the Society for submitting an account of this investigation at so early a stage, but as I may be unable to go further into the matter for some time, I offer these results in the hope that they may prove of some assistance (however slight) to others whose work lies in the same direction.

I have to express my indebtedness to Mr. G. E. Shaw, B.Sc., F.I.C., etc., late senior assistant in the analytical laboratory of my firm, for his valuable help in carrying out the analyses described.

Discussion.

Mr. Chapman, who had read the paper in the author's absence, added that Mr. Howard had been good enough to undertake this matter in connection with the Council's investigation scheme. The object had been to ascertain whether Keller's method could be modified with advantage, as had been stated to be the case by Gordin, who had published a modification of Keller's method, involving the omission of the nitric acid and the substitution for it of a further quantity of much stronger sulphuric acid. The method as modified by Gordin gave, it was claimed, exact results, whereas Keller's method was stated to yield only 96 per cent. of the total strychnine present. Mr. Howard's results, however, went to show that Keller's method was capable of giving not 96 per cent. of the strychnine, but, if anything, rather more than the actual quantity present, and consequently that its modification for the reasons given by Gordin was not necessary.

The President (Mr. Bevan) said this seemed to be one of those cases in which very strict observance of time and temperature conditions was essential.

Mr. F. C. J. Bird said that the method laid down in the British Pharmacopœia for the determination of strychnine in preparations of nux vomica was not described in very full detail, a good deal being left to the judgment of the worker. As a matter of fact, the method was hedged around by conditions, not stated in the text, which it was absolutely necessary to observe in order to get correct results. In a recent case in his own experience several samples of nux vomica preparations had been condemned by certain analysts as containing excessive amounts of strychnine, whereas on further analysis they had been found to contain the correct amount only. Such errors were due simply to want of knowledge of the particular conditions under which the Pharmacopœia process gave correct results. In the precipitation of the strychnine by ferrocyanide of potassium the prescribed strength of sulphuric acid must be strictly observed, and a certain definite amount of wash-water (at 70° F.) used, a correction being made for the amount of strychnine ferrocyanide dissolved by the wash-water. The temperature of the wash-water was also a most important factor. He had himself found that the same sample of a preparation of nux vomica yielded very different results when examined in summer and in winter respectively, the difference being due almost entirely to the variation in the temperature of the wash-water. At low temperatures—*e.g.*, in winter-time—brucine ferrocyanide was precipitated with the strychnine ferrocyanide, and came out in the final weighing as strychnine, making the result too high. In order, therefore, that the Pharmacopœia process might be properly understood, it was necessary that more detailed instructions should be given with regard to it. The method in which the brucine was destroyed by means of nitric acid was a very promising one, and Mr. Howard had placed it on a sound basis in showing that at 0° C. it gave practically the exact amount of strychnine present. As a member of the committee of reference appointed by the Pharmacopœia Committee of the General Medical Council, he could assure Mr. Howard that his results would be welcomed and greatly appreciated by those who were endeavouring to raise the analytical processes of the Pharmacopœia to that high level of accuracy and reliability on which it was most desirable that they should be placed.

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NOTE ON AN OBJECTIONABLE METHOD OF FINING WINES.

By R. BODMER.

(*Read at the Meeting, May* 3, 1905.)

ABOUT six months ago I was supplied with some Moselle (Zeltinger) by a firm of wine merchants. In several bottles I noticed a blue sediment in the last glass poured out.

I collected some of the sediment and examined it. It proved to be a ferrocyanide of iron, and also gave a distinct reaction for zinc. I was at a loss to understand how these substances could be introduced into wine, but on mentioning the matter to Mr. Hehner he showed me a note in the *Zeitschrift für Untersuchung der Nahrungs und Genussmittel* (1903, p. 452), by Dr. Karl Windisch on certain "finings" which he had examined. They were sold as "Heins Schnell-Klärung," and consisted of two solutions:

1. A solution of zinc sulphate—11·417 grams (cryst.) in 100 c.c.
2. A solution of potassium ferrocyanide—10·52 grams in 100 c.c.

The solutions are supposed to be of exactly equivalent strength.

No. 1 is first added to the wine—about 200 c.c. to 200 litres of wine—and then an equal volume of No. 2. A bulky precipitate of zinc ferrocyanide is formed, which, in settling out, rapidly and completely clarifies the wine.

On further inquiry into the matter, I found that these solutions had actually been used by the importers of the wine under the impression that they contained nothing injurious to health and did not contravene the German wine laws. I may say that this preparation is patented in Germany and Sweden, and is advertised by the vendor as being safe and permissible, and the advertisement contains a certificate from a German analyst to the above effect.

On further examining some of the wine, I detected ferrocyanide in solution after the blue precipitate had been filtered off.

I next obtained some more wine of the same character from a totally different source. In this I could detect no ferrocyanide in solution nor was there any blue sediment, but the wine contained zinc. In order to corroborate me, Mr. Hehner kindly examined half of a mixture of two bottles of wine, and I examined the other half. Our results agreed very closely. Mr. Hehner found zinc in the proportion of 27·6 mgms. calculated as crystallized zinc sulphate per litre, and I obtained 28 mgms. The latter figure is equivalent to 1·96 grains per gallon, or 0·32 grains per bottle.

Although this proportion of zinc sulphate is small, it is nevertheless decidedly objectionable in a light wine, of which some persons drink as much as two bottles a day.

This process of fining is a most dangerous one. If the solutions do not exactly balance each other, the wine will contain either an excess of zinc sulphate or potassium ferrocyanide.

I have proved by experiment that traces of hydrocyanic acid are evolved from a wine of the acid character of Moselle to which a little ferrocyanide of potassium has been added.

Further, as shown by Dr. Windisch in the note above referred to, zinc ferrocyanide is not entirely insoluble in an acid wine, so that some of the precipitate first formed may be redissolved.

As a warning to wine merchants and importers, a letter signed by Sir Thomas Stevenson, Mr. Hehner, and myself was sent to four wine and spirit trade papers, pointing out the dangerous nature of this preparation.

As to the method of analysis adopted: As a qualitative test, a few drops of ferrocyanide of potassium was added to some of the wine. A bluish-white precipitate was produced, which on further examination was found to contain zinc. This is, of course, not a conclusive test, as some wines which contain no zinc were found to give more or less precipitate with potassium ferrocyanide, no doubt due to the presence of a little albuminous matter in the wine (from isinglass, used as finings). Still, I noted that the quantity of this precipitate was more decided in wine containing zinc than when none was present. For the quantitative determination 500 c.c. of the wine was evaporated, and the residue charred with nitric and sulphuric acid (as in the method for determining arsenic in beer). The residue was well extracted with boiling water and a little hydrochloric acid.

Ammonia was added in excess (after the iron present had been oxidized by boiling with a little nitric acid) and the precipitate of ferric hydroxide, etc., filtered off. The filtrate was acidulated with acetic acid and a stream of SH_2 passed through for some time. The precipitated zinc sulphide, after washing with SH_2 water, was dissolved off the filter with dilute nitric acid and the solution evaporated, ignited, and the residue weighed as ZnO.

In one experiment I ignited the precipitate of ZnS with the filter-paper, and there was a decided loss, owing, no doubt, to reduction of the ZnO to metallic zinc and volatilization. The ignited residue dissolved in dilute HCl gave copious reaction for zinc with ferrocyanide of potassium in acetic acid solution and with ammonium sulphide in ammoniacal solution.

I have been informed by the wine importers from which this wine was obtained that samples of this identical wine and others in which Mr. Hehner and Dr. Dyer also found zinc were sent to Germany for analysis, and that no zinc was detected in any of them. I ascertained, however, that the wines were incinerated and the ash examined for zinc. Dr. Windisch has pointed out that by this procedure a *loss* of zinc is likely to occur; so this may perhaps account for the fact that the German chemists found no zinc, or else the samples could not have been identical with those examined by Mr. Hehner, Dr. Dyer, and myself.

I have only succeeeded in finding ferrocyanide in solution in one sample—the one first referred to, in which the blue sediment was present, and where the use of these finings was admitted.

A few drops of ferric chloride were added to the wine previously acidulated with a few drops of hydrochloric acid. The blue precipitate—much of which was probably tannate of iron—was filtered off, washed, and boiled with caustic soda solution. The ferric hydrate was filtered off, and the filtrate acidulated with HCl. On adding a drop of ferric chloride a blue precipitate was again obtained.

The mere fact that ferric chloride produces a bluish precipitate with a wine is not conclusive evidence of the presence of a ferrocyanide, as the tannate of iron also has a decidedly bluish tint.

In the paper by Dr. Windisch already referred to he states that "Heins Schnell-Klärung" has been used in Germany to a very considerable extent, and not only for wines but for spirits and liqueurs. I believe, however, that the use of this preparation is now prohibited in Germany, although patented there.

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AMMONIUM OXALATE, ITS FORMULA AND STABILITY.

By P. V. DUPRÉ.

(*Read at the Meeting, June* 7, 1905.)

The introduction of the Act regulating the use of explosives in coal-mines rendered necessary the production of explosives which, while possessing sufficient power for practical purposes, could yet be used with safety, or at least with comparative safety, in coal-mines. In many instances this object was attained by the addition of some ingredient which had the effect of lowering the temperature produced by the explosion. Among such ingredients ammonium oxalate occupies a prominent position.

In order to render possible the accurate analysis of explosives containing this ingredient, a knowledge, not only of its composition, but also of its behaviour under various conditions, became necessary, and as this information could not be obtained from available books of reference, the work which I now beg to lay before the members of this Society was undertaken.

At the outset it appeared that even the composition of ammonium oxalate was given differently in different books; for instance, although the majority gave it as containing one molecule only of water of crystallization, in Tilden's edition of Watts's "Organic Chemistry," published in 1886, it was stated to contain two molecules, while the first edition of Perkin and Kipping's "Organic Chemistry" omitted the water altogether, although this was given for oxalic acid (in the latest edition this has been corrected, one molecule being given).

On this account, although the main object of the work was to determine the behaviour of ammonium oxalate under various conditions, it was considered advisable first to settle the question of its composition.

For this purpose a sample of pure ammonium oxalate was taken and a portion further purified by recrystallization. Known portions were heated in a water-oven at a temperature of about 95° C. until they ceased to lose weight, when the mean of a large number of experiments which showed very slight variations gave a loss of 12·67 per cent. This is the exact value obtained by calculation on the assumption that the salt contains one molecule of water.

In order to see whether the point at which the loss in weight ceased was really definite a portion was subjected to prolonged heating at 94° C. Table I. gives the figures obtained.

TABLE I.

HEATED IN WATER-OVEN AT 94° C.

Time.	Weight.	Loss Per Cent.
No hours	1·666 grams.	—
Two hours	1·454 „	12·72
Seven hours	1·454 „	12·72
Ten hours	1·453 „	12·78
Sixteen hours	1·451 „	12·90
Twenty hours	1·451 „	12·90
Twenty-six hours	1·451 „	12·90

It will be observed, bearing in mind that the theoretical loss of 12·67 per cent. is complete in about half an hour, that the salt suffers no further decomposition for seven hours, and even after twenty-six hours the further loss is very small.

There was, however, the possibility that the loss might not be exclusively that of water. The analysis was therefore completed by the estimation of oxalic acid by means of a carefully standardized solution of potassium permanganate, and of ammonia by distillation, both in the original salt and in a portion of the dried salt. The results are given in the following table, which gives the mean of three closely concordant analyses:

TABLE II.

Name of Constituent.	$(NH_4)_2C_2O_4.H_2O$.		$(NH_4)_2C_2O_4$.	
	Calculated.	Estimated.	Calculated.	Estimated.
NH_3	23·94	23·94	27·42	27·38
$H_2C_2O_4$	63·38	63·38	72·58	72·58
H_2O	12·67	12·67	—	—
	99·99	99·99	100·00	100·00

The analysis thus agrees very closely indeed with the calculated value, and consequently the formula $(NH_4)_2C_2O_4.H_2O$ may safely be assumed to be the correct one.

Experiments were then made with a view to discovering whether under any conditions of crystallization a salt could be obtained with either more or less than one molecule of water. Crystals were prepared at the boiling temperature, at ordinary temperatures, and at a temperature near the freezing-point, the crystals formed being quickly dried by pressure between sheets of blotting-paper and the water estimated as before. In no case were crystals obtained containing more or less than one molecule of water, and it seems certain that if a form with two molecules of water can exist, such a form must be extremely unstable.

Having therefore settled the question of formula, the investigation as to the exact conditions under which the salt loses its water was begun, an accurate knowledge of these conditions being necessary in order to render possible the estimation of moisture in an ammonium oxalate explosive. Weighed portions were put into an air-chamber, kept at constant temperature night and day, and weighed every twenty-four hours for from two to sixteen days. It was found that up to a temperature of 30° C. no loss of weight took place even in eight days; at 40° C. the loss was very small even in four or five days; while at 50° C. the loss in five days only amounted to 2·2 per cent. It thus appears that when heated in ordinary air the oxalate remains stable up to a temperature of from 30° to 40° C., but above this temperature it begins to lose water very slowly.

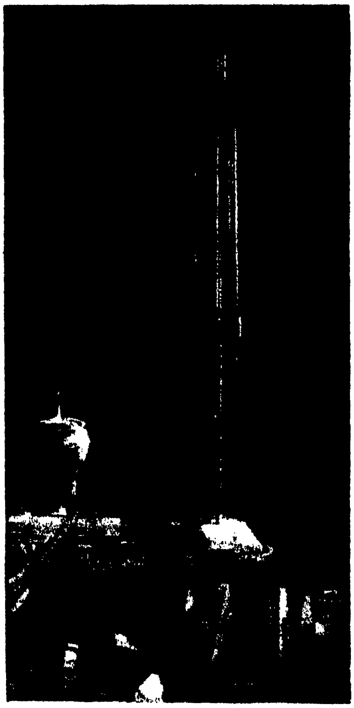
Weighed portions were then placed in a sulphuric acid desiccator, the whole being kept at constant temperature in an incubator. The following table gives the results:

TABLE III.

Temp. 12° C.		Temp. 20° C.		Temp. 30° C.		Temp. 40° C.	
Time.	Loss. Per Cent.	Time.	Loss. Per Cent.	Time.	Loss. Per Cent.	Time.	Loss. Per Cent.
4 days	—	3 days	1·44	1 day	2·51	1 day	7·30
11 ,,	2·23	5 ,,	2·94	3 days	7·62	2 days	12·64
16 ,,	5·35	8 ,,	4·75	4 ,,	10·13		
				5 ,,	11·59		
				6 ,,	12·40		
				7 ,,	12·64		

Thus it appears that in perfectly dry air ammonium oxalate loses water even at 12° C., although but slowly, while at 40° C. the loss is fairly rapid, being complete in two days.

Since the percentage of moisture in explosives is often estimated by drying over sulphuric acid under reduced pressure, it was now necessary to find out the behaviour of ammonium oxalate under similar conditions. The last experiments were therefore repeated, with the difference that the desiccator was exhausted to about 1 millimetre before being placed in the incubator. The results showed that the percentage loss was the same as before, but took place much more rapidly, the salt losing all its moisture in three days at so low a temperature as 44° F., or 6·7° C.

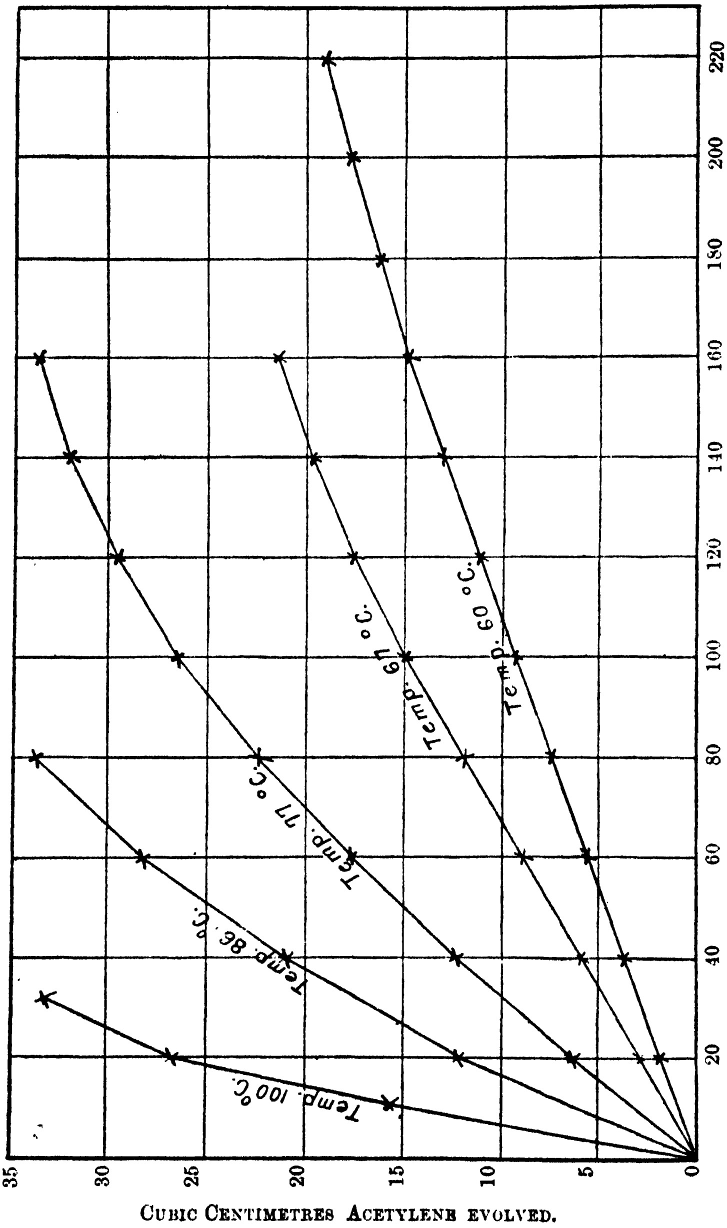


These experiments, however, did not show whether the rate of loss was uniform, nor did they determine accurately the effect on the rate of loss caused by a definite rise of temperature, and in order to do this it was necessary to find some more convenient and accurate method by which the loss could be observed. As it would be very difficult, if not impossible, to measure the water evolved volumetrically, and determination by weighing precluded the possibility of continuous observation, it appeared that if the water vapour evolved could be converted into an equivalent quantity of some gas, measurement would be much more convenient. For this purpose calcium carbide seemed to possess the necessary qualities, and the following experiment was devised. A known weight of oxalate was placed in a small tube, and a quantity of calcium carbide mixed with it, the tube being connected with a nitrometer (see illustration) to measure the acetylene evolved by the action of the water vapour on the carbide, the tube being kept at the required temperature by immersion in an oil-bath. The first result was somewhat startling, for directly the tube was immersed in the oil-bath, which was at a temperature of 70° C., gas was rapidly evolved, the evolution ceasing in about half a minute, when the volume of gas corresponded fairly closely with the theoretical quantity obtainable from the water present in the quantity of ammonium oxalate taken. Obviously the carbide enormously increased the rate of decomposition. In the next experiment, therefore, the carbide was separated from the ammonium oxalate by means of a thin layer of sand, the experiment being otherwise carried out in the same way. The results then obtained were very satisfactory, and are shown in the form of curves (Fig. 1). Experiments are now in hand to ascertain whether this method of estimating moisture can be applied to other substances.

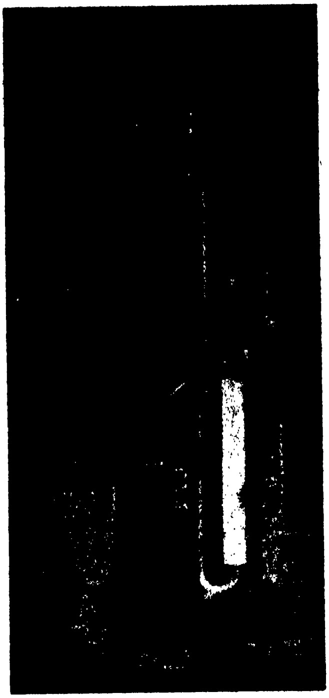
It will be seen that the rate of decomposition is very nearly constant for any given temperature, and approximately doubles for every rise of 10° C.

As it was important in connection with the heat test to ascertain whether even minute traces of ammonia were lost with the water, a small quantity was submitted to the conditions of the heat test, the iodized starch-paper being replaced by moistened red litmus-paper. It was found that after a few minutes the litmus-paper turned blue, showing that some ammonia, at any rate, was given off. This quantity was estimated by means of Nessler solution, and was found to be extremely small, and, as far as analysis is concerned, may be neglected.

FIGURE 1.
TIME IN MINUTES.



It might, nevertheless, be supposed that the presence of ammonium oxalate in an explosive would materially affect the heat test. Experiments made tend to show, however, that, although in the case of an explosive giving a high heat test the effect is very noticeable, it is of no practical importance in the case of samples giving a low test.



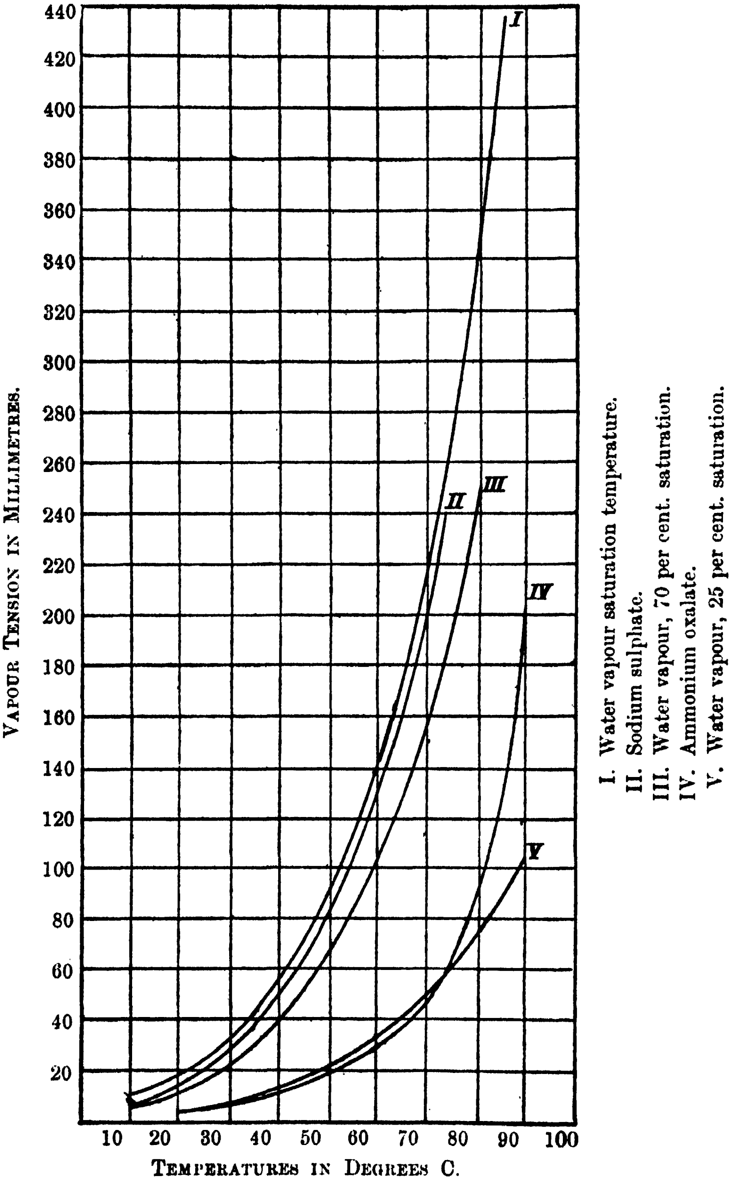
Lastly, as a salt containing water of crystallization does not effloresce in air unless its vapour tension exceeds that of the water vapour present in the air, it was thought that the determination of the vapour tension of ammonium oxalate at various temperatures might give valuable indications of the stability of the salt under varying conditions of temperature. As a rather wide range of pressure—about 300 mm. of mercury—was required, the apparatus used was as follows: As shown in the figure, it consisted of a U-tube, one limb of which was slightly longer than the maximum barometric height, the other about half as long, this latter being graduated in millimetres. The top of the long limb was closed by a stopper fitted with a tap, so that the tube could be exhausted by means of an air-pump or Sprengel pump, if desired. Such a quantity of mercury was put into the tube that, when the longer limb was completely exhausted, the mercury stood at or a little above the zero mark on the shorter limb. The upper half of the longer limb passed through a water-jacket, which could be kept at any desired temperature. For an experiment, the mercury was first forced up into the longer limb by means of an air-pump connected with the shorter limb to within a few centimetres of the stopper; a small quantity of ammonium oxalate—about 2 grams—was then introduced into the longer limb, the stopper replaced, and the tube completely exhausted by means of a vacuum-pump. The reading of the surface of the mercury in the shorter limb was then taken, and the temperature of the water-jacket slowly raised, readings being taken every 5° or 10°.

These results are plotted in the form of a curve (Fig. 2). Curves for the tension of the aqueous vapour in air—when saturated, 70 per cent. saturated, which may be taken as the average hygrometric condition of the air, and 25 per cent. saturated—are also given; and, lastly, a vapour tension curve for sodium sulphate.

The curves show that the vapour tension of ammonium oxalate does not reach that of water even at a temperature of 95° C., nor does it even reach the 70 per cent. curve; in fact, the salt will be stable at all temperatures up to at least 95° C., provided the air is not less than half saturated with moisture. The curve for sodium sulphate, on the other hand, is always above the 70 per cent. curve, showing that the salt will effloresce at all temperatures in ordinary air—a fact proved by practical experience.

It will be noticed that the 25 per cent. saturation curve cuts that of ammonium oxalate in two points between 60° and 70° C.; hence, between these temperatures the salt would appear to be stable in air only 25 per cent. saturated, while outside these it would lose water.

FIGURE 2.



Lastly, the curve for ammonium oxalate may be used to determine the temperature to which the salt must be heated in a water-oven in order that it may lose water. For the tension of the aqueous vapour inside the oven will probably be approximately the same as that outside; hence, this tension being known, it is simply necessary to find out at what temperature the oxalate curve reaches this value. For example, take the temperature of 20° C., the average tension of aqueous vapour will be 9 millimetres, and the ammonium oxalate curve is found to reach this value at about 36° C.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Ammonia in Milk, and its Value as a Test of Purity. A. Trillat and Sauton. (*Bull. Soc. Chim.*, 1905, xxxiii., 719-723.)—The method of determining nitrogen, based upon the formation of nitrogen iodide (ANALYST, xxx., 218), can be applied to milk after separation of albuminoid matters, which would otherwise combine with the iodine. The simplest method is to treat the milk with iodine trichloride, which also reacts with traces of ammonia to form nitrogen iodide when the liquid is rendered alkaline. For this purpose 10 c.c. of the milk are treated with 10 c.c. of a 10 per cent. solution of iodine trichloride, and immediately filtered, and the filtrate treated little by little with pure milk of lime (2 to 3 parts of lime to 100 of water) until (in the presence of ammonia) an intense black precipitate of nitrogen iodide, which disappears on adding an excess of the reagent, is formed. The method is capable of detecting 1 part of ammonia in 100,000 of milk, and a quantitative determination can be made by colorimetric comparison with standard samples.

In determining the value of this reaction as a criterion of the purity of milk, the authors made experiments on samples of pure milk, whole, diluted, and sterilized, and on milk watered with impure water, and infected with different pathogenic and other organisms. It was found that no ammonia was produced by acetic or lactic bacteria, or by the bacilli of typhoid fever, anthrax, or tuberculosis. On the other hand, ammonia was produced by *Micrococcus ureæ*, certain species of *tyrothrix*, and Flügge's peptonizing bacillus, which is of frequent occurrence in milk. The following results are given to show the progress of the formation of ammonia in sterilized milk inoculated with different micro-organisms. In each case the acidity of the milk is expressed in terms of decinormal sodium hydroxide solution per 10 c.c. of milk and the ammonia in mgms. per litre.

Observations taken after	Control Sample.		Micrococcus Ureæ.		Tyrothrix Tenuis.		Tyrothrix Filiformis.		Flügge's Bacillus.		Bacteria of Sewage Effluent.		Bacteria of Decomposed Meat Juice.		Bacteria of Decomposed Urine.	
	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.	Acidity.	NH_3.
Four hours	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0
Eight hours	1	0	1	0	1	0	1	0	1	0	1·3	0	1	0	1	0
Sixteen hours	1	0	1	trace	1·5	0	1·5	0	1·5	0	2·9	0	1·3	0	1	0
Twenty-four hours ..	1	0	1·3	25	1·8	0	2·2	0	2	0	3·3	16	1·8	20	1·3	20
Thirty-six hours ..	1	0	5·6	33	4·6	20	5	20	4·8	20	6·4	25	4·3	22	2·3	22
Seventy-two hours ..	1	0	9	50	5·9	25	6	25	6	25	12	30	6·2	25	4·5	33

Two factors were found to play a part in the rate of formation of ammonia—the temperature and mode of inoculation. When the inoculation was made by means of a platinum wire, milks kept at 15° to 20° C. coagulated before the appearance of ammonia, while the reverse was the case when the same milks were kept at 35° C. But if the inoculation was on a larger scale the ammonia reaction was obtained long before the coagulation of the milk kept at the ordinary temperature. It was proved experimentally that milk watered with 10 per cent. of impure (Seine) water showed the reaction before coagulation, and that the amounts of ammonia often reached 20 to 25 mgms. per litre. These results were confirmed by the analysis of milks found to be watered in the Paris Municipal Laboratory. In each case pronounced reactions for ammonia were obtained. Milks infected with sewage effluent (2 drops per litre) also gave the ammonia reaction before coagulation. The general conclusions drawn by the authors are that healthy cow's milk which has not been exposed to improper conditions—*e.g.*, in badly ventilated stalls—ought not to contain ammonia, and that if it is present in any considerable quantity it should be regarded as raising a presumption of either pollution or watering. C. A. M.

Determination of Free Sulphuric Acid in Vinegar. C. Rossi. (*L'Industria Chim.*, 1904, vi., 253, 254; through *Zeit. Untersuch. Nahr. und Genussmittel*, 1905, ix., 698, 699.)—Whilst acetic acid in aqueous solution has an acid reaction towards methyl orange, in the presence of alcohol or acetone it is quite indifferent to this indicator. Free sulphuric acid in vinegar can, therefore, be directly titrated if alcohol or acetone be previously added and methyl orange used as indicator. To each 10 c.c. of vinegar at least 6·5 c.c. of alcohol or acetone should be added (*cf.* Schidrowitz, ANALYST, xxviii., 233). W. P. S.

The Determination of Acetyl-Methyl-Carbinol in Vinegar. Pastureau. (*Journ. Pharm. Chim.*, 1905, xxi., 593-595.)—Several samples of vinegar examined by the author gave abundant precipitates on the addition of 95 per cent. alcohol, and strongly reduced Fehling's solution in the cold. From one of them, methyl acetol, or acetyl-methyl-carbinol,

$$CH_3.CO.CH(OH).CH_3,$$

was isolated by neutralizing the vinegar with sodium carbonate and subsequently distilling. The distillate gave an abundant precipitate of iodoform on treatment with iodine in an alkaline medium, and yielded with phenylhydrazine a yellow osazone melting at 243° C. The solution of this osazone gave a blood-red coloration on the addition of a trace of ferric chloride, and on evaporation yielded red acicular crystals.

In the determination 50 c.c. of the vinegar were neutralized with sodium carbonate and distilled to dryness, superheating being avoided. The distillate was rendered alkaline and treated with 10 c.c. of a $\frac{N}{10}$ solution of silver nitrate. After standing for twenty-four hours, it was diluted to 100 c.c. and filtered, and the residual silver determined by the cyanometric method. The amount of acetyl-methyl-carbinol was then calculated in accordance with the equation:

$$3(CH_3.CO.CHOH.CH_3) + AgNO_3 = 3(CH_3.CO.CO.CH_3) + 3H_2O + N + Ag.$$

In one experiment 0·0674 gram of silver was found to have been reduced, and this corresponded to 0·1628 gram of acetyl-methyl-carbinol in the 50 c.c. of vinegar, or 3·256 grams per litre. The vinegars were grain products, and in the author's opinion the acetyl-methyl-carbinol was produced by the action of bacilli of the *tartricus* species on the carbohydrates. C. A. M.

Determination of Vanillin, Coumarin, and Acetanilide in Vanilla Extract. A. L. Winton and E. Monroe Bailey. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 719-724.)—The slight loss of coumarin which was found to take place during the determination of vanillin, etc., according to the method previously described by one of the authors (ANALYST, xxviii., 37), is obviated in the following modification of that method. The loss was sustained during the removal of the alcohol from the extract. Twenty-five grams of the latter are placed in a beaker with marks showing volumes of 25 and 50 c.c. Water is added up to the 50 c.c. mark, and the mixture evaporated at a temperature below 70° C. to a volume of 25 c.c. This dilution and evaporation is once again repeated. Normal lead acetate solution is then added drop by drop, with stirring, until no further precipitate forms. The precipitate is collected on a moistened filter, and washed three times with hot water, taking care that the total filtrate does not exceed 50 c.c. The filtrate is cooled and shaken out four times with ether, using 15 c.c. each time. The combined ethereal extracts are now shaken out with four or five successive quantities of 2 per cent. ammonia, and the ammoniacal solutions set aside. After transferring the ether solution to a weighed dish, it is allowed to evaporate at the ordinary temperature, dried in a desiccator, and weighed. The residue is stirred up three times with 15 c.c. of light petroleum (B.P. 30° to 40° C.) and decanted, thus removing the coumarin. The residue in the basin is dried and re-weighed, to obtain weight of the coumarin extracted, whilst the residue itself, if acetanilide, should have a melting-point of about 112° C., and respond to the qualitative tests for this substance. The light petroleum extract is allowed to evaporate at the ordinary temperature. If the residue be pure coumarin it should melt at 67° C. The ammoniacal solutions are now acidified with hydrochloric acid, cooled, and shaken out four times with ether, the ethereal extracts evaporated at the ordinary temperature in a weighed platinum dish, dried over sulphuric acid, and weighed. The residue, consisting of pure vanillin, should melt at about 80° C. If, however, acetanilide has previously been detected, this residue is dissolved in 15 c.c. of 10 per cent. ammonia and shaken out twice with an equal volume of ether. By evaporating the ethereal solutions and weighing the residue, the amount of acetanilide in the vanillin is obtained. The total quantity of acetanilide is found by adding the weight of this last extract to that of the residue previously obtained, and identified as acetanilide. Analyses of vanilla extracts, containing known amounts of vanillin, coumarin, and acetanilide, show that the above process of separation is trustworthy, the error in no case exceeding 0·02 per cent. on the quantities taken. W. P. S.

Detection of Antipyrin in Pyramidon. P. Bourcet. (*Bull. des Sci. Pharm.*, vii., 218; through *Pharm. Journ.*, 1905, lxxiv., 845.)—As little as 2 per cent.

of antypyrin in pyramidon (dimethyl-amido-dimethyl-oxyquinizine) may be detected by the following reaction: About 0·02 gram of the sample is dissolved in 5 c.c. of cold water and shaken with 2 c.c. of sulphuric acid and 2 drops of saturated sodium nitrite solution (or a little of the salt). Pyramidon gives an intense bluish-violet coloration, which rapidly fades, leaving the solution colourless; if antipyrin be present, a stable bluish-green colour is perceptible after the bluish-violet, due to the pyramidon, has faded away. W. P. S.

Volumetric Determination of Mercury in Mercuric Salicylate. E. Ruff and P. Nöll. (*Archiv. d. Pharm.*, ccxliii., 1; through *Pharm. Journ.*, 1905, lxxiv., 821.)—The following method, which is stated to be rapid and accurate, is also applicable to other organic mercurial compounds. 0·3 gram of the mercuric salicylate is heated with 4 grams of potassium sulphate and 5 c.c. of concentrated sulphuric acid in a 150 c.c. flask, resting on wire gauze, and provided with a reflux tube about 50 centimetres long. As soon as the mixture is clear and colourless, the reflux tube is rinsed out with from 5 to 10 c.c. of sulphuric acid; about 0·2 gram of potassium permanganate is added to the contents of the flask, and the heating continued until the solution is again colourless. After diluting to 100 c.c., the solution is titrated with $\frac{N}{10}$ sulphocyanide solution, using iron-alum as indicator, until a reddish brown colour appears. One c.c. of $\frac{N}{10}$ sulphocyanide is equivalent to 0·010015 gram of mercury. W. P. S.

ORGANIC ANALYSIS.

A Test of the Purity of Cocoanut Oil. E. Milliau. (*Comptes Rend.*, 1905, cxl., 1702, 1703.)—The presence of seed oils in cocoanut oil may be detected by the gooseberry-red colour produced by the simultaneous reaction of phloroglucinol and resorcinol in an acid medium. Four c.c. of the clear dry sample are shaken with 2 c.c. of a freshly-prepared saturated ethereal solution of phloroglucinol and then with 2 c.c. of a freshly-prepared saturated solution in benzene of resorcinol, and the tube cooled for a few moments in water at 10° C. The contents are then mixed with 4 c.c. of nitric acid S.G. 1·38, transferred to another tube, and vigorously shaken for five seconds. Pure cocoanut oil shows little or no alteration, while negligible traces of impurities give a very faint rose tint, which rapidly disappears. But if a seed oil or lard oil, oleonaphtha or resin oil, be present in the proportion of 5 per cent. or even less, the characteristic red coloration immediately appears. No notice is taken of reactions subsequently produced under the prolonged action of the nitric acid. C. A. M.

The Characteristics of Sperm Oil. G. Fendler. (*Chem Zeit.*, 1905, xxix., 555, 556.)—A specimen of undoubted purity yielded 15 per cent. of spermaceti, which, after purification, gave the following values: Specific gravity at 15° C., 0·942; melting-point, 42° C.; saponification value, 134; iodine value (Hübl), 9·3; unsaponifiable substances, 51·07 per cent., melting at 45° C.

The oil separated from the spermaceti gave the following results: Specific gravity at 20° C., 0·8781; solidification-point, 15·5° C.; melting-point, 18·0° C.;

Reichert-Meissl value, 0·60; saponification value, 150·3; iodine value, 62·2; and unsaponifiable matter (alcohols), 39·17 per cent. The fatty acids formed a brownish-yellow oily liquid with the following characteristics: Specific gravity at 15° C., 0·8999; solidification-point, 12·4° C.; melting-point, 18·8° C.; acid value (saponification value), 236·2; iodine value, 68·64; acetyl value, 17·9; acetyl acid value, 222·5; mean molecular equivalent, 237·7; liquid fatty acids, 85·78 per cent.; solid fatty acids, 14·22 per cent. The solid fatty acids separated by the lead method melted at 38·2° C., and had acid value of 242·5, and mean molecular equivalent of 231·6; whilst the liquid fatty acids had an acid value of 228·7, a mean molecular equivalent of 245·6, and an iodine value of 75·6. The liquid acid distilled under reduced pressure between 195° and 250° C. yielding fractions, of which the highest molecular equivalent was 287·4. The presence of physetoleic acid appeared doubtful in the light of these determinations.

Contrary to the experience of Allen and Lewkowitsch, glycerin was found, 800 grams of the oil yielding 14·2 grams of thick brown syrup containing 74·33 of pure glycerin, corresponding to 1·32 per cent. in the original oil. According to Benedikt and Ulzer's assertion, this would represent adulteration with 13·2 per cent. of a fatty oil, whereas the purity of this sperm oil, obtained direct from the captain of the ship, was beyond question. C. A. M.

Colour Reactions of Carbohydrates. R. and O. Adler. (*Arch. Physiol.*, 1905, cvi., 323; *Chem. Zeit. Rep.*, 1905, xxix., 121.)—The furfural test for pentoses can be simplified by decomposing them with acetic acid instead of employing the usual tedious distillation with hydrochloric acid. If a boiling mixture of equal parts of glacial acetic acid and aniline be treated with a few drops of a solution of a pentose or a fragment of the solid sugar, the red coloration of furfural-aniline acetate soon appears. Methyl pentoses (rhamnose) react in an analogous manner, yielding the corresponding methyl furfural amines; but carbohydrates, other than pentoses, do not yield the slightest trace of furfural within so short a time, with the exception of heptoses, which give the reaction. Other amines that form similar compounds with furfural may be used instead of aniline in this test. Certain phenols also give intense colorations when heated with sugars in the presence of acetic acid and some hydrochloric acid. The resorcinol reaction for fructose is equally conclusive when acetic acid and one or two drops of hydrochloric acid are used; whilst Tollens' tests for pentoses by means of orcinol and phloroglucinol give satisfactory reactions when carried out in this way, provided a few drops of hydrochloric acid be added to the acetic acid. Hexoses and di- and tri-saccharides that yield hexoses behave in quite a different manner. If a small quantity of dextrose be heated with the mixture of aniline and acetic acid, a reddish-brown colour is produced, which changes, on standing, to a bright green. This reaction is also given by the aldoses: mannose and galactose; by the ketoses: fructose and sorbinose; and by di-, tri-, and polysaccharides after preliminary decomposition. Other amines can be used in place of aniline. The green colouring matter formed is insoluble in water, but dissolves in ether. C. A. M.

Investigation of Tollens' Phloroglucinol Reaction for Pentoses. E. Pinoff. (*Berichte*, 1905, xxxviii., 766.)—If the test for pentoses by means of phloroglucinol and hydrochloric acid be made in an alcoholic instead of an aqueous solution, the coloration remains stable for a week if kept in diffused light and with the addition of ether. The spectrum shows one, two, or three adjacent absorption bands, according to the proportion of the three active agents in the test, and these bands are probably characteristic of three separate compounds. Evidence in support of this is that it is possible to prepare three distinct solutions, each of which shows only one of the bands. The band characteristic of pentoses is in the yellow part of the spectrum, and on heating the solutions showing the other bands with hydrochloric acid, they too yield liquids which also show only the pentose band. C. A. M.

Colour Reactions of Pyruvic Acid with α- and β-Naphthols in Sulphuric Acid Solution. P. Alvarez. (*Bull. Soc. Chim.*, 1905, xxxiii., 716, 717.)—The reagents used are freshly prepared solutions of 0·02 to 0·05 gram of α- or β-naphthol in 1 c.c. of sulphuric acid of specific gravity 1·83. One drop of the organic acid is added to 10 drops of the reagent in a porcelain crucible, which is then slightly warmed over a spirit-lamp. (i.) *β-Naphthol Reagent:* A bright-red colour is immediately produced, and on gently warming the liquid it becomes a very intense blue, which changes to a fugitive yellow on the addition of water or concentrated alcohol. (ii.) *α-Naphthol Reagent:* The colour is yellow in the cold, becoming a very intense orange on heating. On shaking the liquid it adheres to the sides of the crucible like a coloured varnish. The colour does not change on the addition of water or alcohol. These reactions will distinguish pyruvic acid from citric, tartaric, malic acids, etc., and also afford an easy means of distinguishing between α- and β-naphthols. C. A. M.

The Valuation of Lubricants, with Special Reference to Cylinder Oils. F. W. Richardson and H. Norman Hanson. (*Journ. Soc. Chem. Ind.*, 1905, xxvii., 315.)—The paper is one which should be read in full, as it is almost impossible to give an adequate account of the work in a reasonable space. The following is a short summary of the main conclusions:

The authors find that mechanical tests are of little value, a point with which the engineers who assisted in the investigations were compelled to agree. With regard to physical tests, the customary determinations of gravity, viscosity (usually at 210° F.), and flash-points (open and close tests) were not entirely satisfactory. The specific gravity is of very little value, whilst the flash-points are chiefly useful for insurance purposes.

With regard to the viscosity, the authors find that from 500° to 600° F. all cylinder oils have a practically identical value. On the other hand, the figures for 210° or 250° F. have little bearing on actual practice, owing to highly superheated steam being now generally employed.

The authors therefore investigated more closely the value of the surface tension, or, as they suggest it may be termed, the "wetting power" of the oil in thin films in hot air and in steam. Several methods were tried, but none were entirely satisfactory. Finally, the authors determine the viscosity of the oil at 210° before and after exposing for four hours to the action of a current of hot air at 400° F., in a special oven, the oil being spread out in a thin layer. For details of the experimental working, reference should be made to the original paper. The heating for four hours may seem excessive, but the authors point out that in the cylinder the oil makes a rapid succession of film surfaces, and the steam is not only at a very high temperature, but is under high pressure.

They also find that much the same change is produced by steam at 400° as by air at the same temperature, and conclude that the changes are therefore due to polymerization rather than oxidation.

The relative viscosities before and after heating only are used; the absolute values are not calculated.

An interesting point brought out by the experiments is the close relation between the viscometric and the refractometric changes. E. K. H.

The Colorimetric Determination of Adrenaline. J. E. Abelous, L. A. Soulié, and G. Toujan. (*Bull. Soc. Chim.*, 1905, xxxiii., 576.)—The method consists in matching the tint given by adrenaline on the addition of a very dilute solution of iodine. The standard solution is prepared by mixing 10 c.c. of a solution of adrenaline (0·01 per cent.) with 5 c.c. of $\frac{N}{10}$ iodine solution, and allowing it to stand for fifteen minutes at the ordinary temperature, after which a few drops of starch paste are added, and the excess of iodine removed by means of $\frac{N}{10}$ sodium thiosulphate solution. The liquid is then diluted to 50 c.c., and furnishes a rose-coloured standard containing 1 mgm. of adrenaline. In determining the adrenaline in suprarenal glands, 10 grams of the sample are ground up with sand, and the mixture treated little by little with salt water (7 per cent.) previously boiled, and then kept at a temperature of 40° to 50° C., until 100 c.c. in all have been added. The liquid is now slightly acidified with a few drops of a 10 per cent. solution of hydrochloric acid, boiled for a few moments, filtered, and the residue extracted with boiling water until the filtrate amounts in all to 150 c.c. Ten c.c. of the cooled liquid are now treated with iodine as above described, and the rose coloration compared with that of the standard solution. By this method the author found 1·47 mgm. of adrenaline in 1 gram of the suprarenal capsule of a sheep. As the standard solutions of iodized adrenaline lose their colour on keeping, it is advisable to prepare artificial standards of greater stability, such as, for example, tincture of turmeric, reddened by acid and diluted to match the original standard.

C. A. M.

Carbon Tetrachloride as a Solvent for Differentiating Bitumens. Clifford Richardson and C. N. Forrest. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 310.)—The use of carbon tetrachloride as a solvent has been facilitated by the introduction of a much purer commercial article. The authors used, in particular, carbon tetrachloride manufactured by the Acker Process Company, of Niagara Falls, New York, which is almost free from bisulphide.

Carbon tetrachloride, as a general extractive agent, has many advantages. It has a high solvent power, mixes with all other solvents of its class, and dissolves only a trace of water. It is, moreover, non-inflammable and non-explosive, in marked contrast to carbon bisulphide, which ignites at 149° C., and has a considerable effect in making other inflammable solvents non-inflammable when mixed with them.

It has, however, a high specific gravity, and, weight for weight, is not so economical as other solvents.

As regards bitumens, though it is less rapid in its action than carbon bisulphide, it is valuable, since it exercises a selective action on the hydrocarbons and their derivatives which occur in native bitumens, whilst the true asphalts are dissolved by it to the same extent as by carbon bisulphide. It can thus often show the extent to which a native bitumen has been weathered, and, further, the changes which may have been caused by the use of too high temperatures in the industrial preparation of pitches, etc.

The authors recommend that for these purposes the carbon tetrachloride should be used at a temperature not exceeding 25° C., and should be quite free from carbon bisulphide. E. K. H.

Note on Simplified Ultimate Analysis. E. Lippmann. (*Chem. Zeit.*, 1905, xxix., 487.)—The author claims that his method of analysis in which the combustion is aided by copper oxide asbestos (*Chem. Zeit.*, 1903, xxvii., 810) is simpler and cheaper than that of Dennstedt, whilst equally accurate (*vide* ANALYST, this vol., p. 135). The author has succeeded in getting very accurate results even with such a substance as carbon bisulphide, the actual figures being: 0·1663 gram of carbon bisulphide yielded 0·0975 gram CO_2, which corresponds to 15·98 per cent. of carbon, against the calculated 15·78 per cent. E. K. H.

INORGANIC ANALYSIS.

The Analysis of Cupric Ferrocyanide. A. Leuba. (*Ann. de Chim. anal.*, 1905, x., 218, 219.)—When cupric ferrocyanide is boiled with a solution of sodium hydroxide, sodium ferrocyanide is formed and cupric oxide precipitated. This latter dissolves partially in the excess of alkali solution, and if the undissolved oxide be brought into solution by means of nitric acid, the two solutions cannot be mixed, hence the copper in each must be determined separately. This is obviated in the following method, based upon the reaction between cupric ferrocyanide and oxalic acid:

$$Cu_2Fe(CN)_6 + 3H_2C_2O_4 = FeC_2O_4 + 2CuC_2O_4 + 6HCN.$$

From 0·3 to 0·5 gram of the finely powdered ferrocyanide is boiled for about two hours with a 5 per cent. solution of oxalic acid in a flask under a reflux condenser. The resulting yellow precipitate of copper oxalate is washed and dissolved in dilute nitric acid, and the solution added to the filtrate. This is concentrated on the water-bath, and the copper and iron separated by the usual methods. The original solution contains the ferrous oxalate, which is soluble in excess of oxalic acid. It is easily decomposed by nitric acid, and the two nitric acid solutions can be mixed without fear of reprecipitating the original ferrocyanide. Oxalic acid is not suitable for the decomposition of other metallic ferrocyanides. C. A. M.

A Method of Separating Arsenic. H. Cantoni and J. Chautems. (*Ann. de Chim. anal.*, 1905, x., 213, 214.)—When a current of dry air at the ordinary temperature is passed over the surface of a solution of arsenious anhydride in concentrated hydrochloric acid to which methyl alcohol has been added, the arsenic is completely volatilized, probably in the form of a methyl arsenious ester. Thus, if a current of air be drawn through a distillation flask, the outlet tube of which is connected with a condenser whose delivery tube is drawn out and dips into a solution of sodium hydroxide, no trace of arsenic is left in the flask. Antimony is not removed under the same conditions. The method should be particularly valuable in toxicological work, since all boiling is obviated. C. A. M.

The Quantitative Determination of Antimony as Trisulphide, and its Separation from Tin. G. Vortmann and A. Metzl. (*Listy Chemické*, 1905, xxix., 65; *Chem. Zeit. Rep.*, 1905, xxix., 120.)—The method is based upon the fact that antimony trisulphide can be converted into the black crystalline modification by means of a current of hydrogen sulphide passed into the hot liquid containing a large amount of hydrochloric acid. The solution of the antimony salt is treated with hydrochloric acid (24 c.c. to 100 c.c.), heated on the water-bath, and frequently shaken while a current of hydrogen sulphide is passed through it. After thirty to thirty-five minutes the conversion of the sulphide into the black form will be complete, and the liquid is diluted with an equal volume of water, and the passage of the gas continued for another two or three minutes. The precipitate is collected in a Gooch's crucible, washed with water and alcohol, and dried at 270° to 280° C. in a current of carbon dioxide. The sulphide may also be converted into pentoxide by igniting it with a weighed quantity of a mixture of ferric oxide and ferric nitrate.

Separation of Antimony from Tin.—The mixed sulphides are dissolved in hydrochloric acid, the solution neutralized with sodium hydroxide, mixed with an equal volume of phosphoric acid (specific gravity 1·30), and after the addition of hydrochloric acid (20 c.c. per 100 c.c.), heated to 100 c.c., and treated with a current of hydrogen sulphide, introduced at first rapidly, and then more slowly. In this way the antimony is precipitated as the crystalline trisulphide, while the tin remains in solution. The filtrate from the precipitate is nearly neutralized with sodium hydroxide, diluted with water, and the tin precipitated from the hot solution by means of hydrogen sulphide. C. A. M.

Determination of Manganese as the Green Sulphide. J. C. Olsen, E. S. Clowes, and Wm. O. Weidmann. (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1622.)—In determining manganese, the authors have obtained very good results by precipitating and weighing it as the green sulphide. This sulphide may be obtained by proceeding as follows: 10 c.c. of the manganese solution, containing 0·15 gram of manganese, are poured into 90 c.c. of a boiling solution of 25 c.c. of ammonium sulphide and 10 c.c. of ammonium chloride solution (containing 2·75 grams ammonium chloride per litre). The ammonium sulphide is prepared by diluting concentrated ammonia with twice its volume of water, saturating half of the liquid in the cold with hydrogen sulphide, and then adding the other half; it must be free from the yellow sulphide. The precipitate is digested in the hot solution for thirty minutes, when it will have been completely converted into the green sulphide. This is filtered off, washed with water containing ammonium sulphide and chloride, dried and ignited as for copper in Rose's method. In the above procedure, it is essential that the manganese solution be poured into the ammonium sulphide, and not *vice versâ*, and that a large excess of ammonium sulphide be present, as otherwise conversion into the green sulphide may not be complete, if it take place at all. A. G. L.

On the Solubility of Oxides of Iron in Hydrofluoric Acid. Ernst Deussen. (*Zeits. angew. Chem.*, 1905, xviii., 813.)—A number of comparative determinations of the solubility of iron rust and scale in hydrochloric, hydrofluoric and oxalic acids showed that dilute hydrofluoric acid dissolves oxides of iron much more easily than do the other acids. The cleaning of rusty iron utensils was also most easily accomplished by means of hydrofluoric acid, the metal itself suffering very little. Iron stains could be easily removed from linen without injuring the fabric, and copper articles were very readily cleaned by dilute hydrofluoric acid, the metal itself being insoluble in the acid.

To remove rust from iron it is recommended to first remove grease, if present, by hot sodium hydrate or carbonate, and then to immerse the vessel for from one to twelve hours in 2 to 5 per cent. hydrofluoric acid contained in wooden vats tarred on the inside. Any unrusted places may be protected by grease or varnish. After removal from the acid the iron is to be washed with hot water and then immediately placed in soda solution or lime-water.

The author mentions that it is possible to immerse one's hands in 5 per cent. hydrofluoric acid for a considerable time without experiencing any ill effects. A rabbit into whose stomach 50 c.c. of 0·5 per cent. pure hydrogen fluoride was introduced showed no signs of poisoning. A. G. L.

Quantitive Determination of Iron and Aluminium in Ignited Mixtures containing large amounts of Al_2O_3 and small amounts of Fe_2O_3. Ernst Deussen. (*Zeits. angew. Chem.*, 1905, xviii., 815.)—The finely divided oxides are intimately mixed with acid potassium fluoride in a platinum crucible. The mixture is fused over a small flame until, after a few minutes, it becomes solid. Dilute sulphuric acid is then added, and the greater part of the hydrofluoric acid expelled by heating for a short time. The sulphates are then dissolved in water in a platinum dish; the iron is reduced by sulphur dioxide, the excess of which is expelled by carbon dioxide, the liquid being then titrated with permanganate in a Jena glass beaker. In one analysis by this method, in which 0·007 gram Fe_2O_3 was present in 0·094 gram of mixed oxides, 0·0065 gram Fe_2O_3 was found. Some experiments are also given to show that the presence of considerable quantities of potassium hydrogen fluoride does not interfere with the titration. The advantages claimed for this method over that of fusing with potassium bisulphate are that no platinum passes into solution, and that it requires less time. A. G. L.

A Case of Iron Contamination in a Water-Supply due to Bacteria. A. Beythien. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 529-531.)—The water-supply of a small town in Saxony was found to contain a large quantity of ferruginous matter in suspension, although when drawn from the well the water was free from iron. Between the well and the town the water passed through about 2 kilometres of asphalted iron mains. An examination of the suspended matter showed that it consisted of a mass of threads similar to *Crenothrix*, incrusted with oxide of iron. These threads were in all probability *Gallionella ferruginea*, and the contamination of the water was due to their presence, aided by the large amount of free carbon dioxide naturally contained in the water. W. P. S.

Method for the Analysis of Bauxite. H. Lienau. (*Chem. Zeit.*, 1905, xxix., 584, 585.)—In view of the increasing manufacture of aluminium from bauxite, the following procedure for the analysis of this mineral is recommended:

Moisture and Combined Water.—Several quantities of about 10 grams each of the same sample are dried at a temperature of 110° C. to obtain the moisture. The combined water is found by heating 1 gram of the dry sample in a platinum crucible in a muffle-furnace. The loss in weight should, more correctly, be termed the loss on ignition, as some specimens contain as much as 1 per cent. of organic matter.

Silica.—One gram of the finely-powdered dry mineral is quickly mixed in a beaker with 8 c.c. of sulphuric acid (1 : 2). After again adding an equal quantity of the acid, the mixture is rapidly brought to boiling, and kept at this temperature until the colour changes from red to yellow or white. The solution is now evaporated until sulphuric acid fumes are evolved. If the latter do not appear before the mixture becomes dry, more acid must be added. Finally, the whole of the free acid is driven off, and the residue, after cooling, boiled with 20 c.c. of dilute sulphuric acid (1 : 5). The solution is diluted with water, and the silica filtered off. If less than 10 per cent. of silica be present, this method of separation is sufficient. With more than 10 per cent., the silica found should be fused with potassium hydrogen sulphate.

Titanic Acid.—The filtrate (or filtrates) from the silica is treated with about 500 c.c. of 3 per cent. sulphurous acid solution, then nearly neutralized with 10 per cent. sodium hydroxide solution, diluted to about 900 c.c., and heated to boiling for one hour. The precipitated titanic acid is collected on a filter, if necessary pouring the first 200 c.c. of filtrate once more through the filter, and washed with hot water containing ammonium chloride.

Iron and Alumina.—The filtrate from the titanic acid precipitate is concentrated over a naked flame and made up to a volume of 500 c.c. In 100 c.c. of this solution the iron is titrated in the usual manner with permanganate solution, after oxidizing and again reducing. A second quantity of 100 c.c. of the solution is oxidized, and the iron and alumina precipitated as hydroxides. W. P. S.

The Determination of Calcium and Magnesium. E. Maigret. (*Bull. Soc. Chim.*, 1905, xxxiii., 631-634.)—The method is based upon the insolubility of magnesia in solutions of sodium chloride containing not less than 160 grams per litre in the presence of sodium hydroxide in the proportion of 0·8 gram per litre, and of the considerable solubility of lime in the same medium.

Determination of Magnesium.—One hundred c.c. of the solution are mixed in a litre-flask with 10 c.c. of a solution of sodium hydroxide (80 grams per litre), previously freed from carbonic acid by the addition of barium chloride in slight excess, and standardized on bi-normal hydrochloric acid. After thorough shaking the liquid is made up to the mark with sodium chloride solution (160 grams per litre) and filtered, and 500 c.c. of the filtrate titrated with the standard acid, with phenol-phthalein as indicator. The difference between twice the amount consumed, and that required by the original 10 c.c. of sodium hydroxide solution, multiplied by 0·95, gives the amount of magnesium chloride per litre.

Determination of Calcium and Magnesium.—One hundred c.c. of the solution are mixed with 20 c.c. of a solution containing 100 grams of sodium carbonate and 20 grams of sodium hydroxide per litre, and standardized on bi-normal acid, using tropaeolin as indicator. The mixture is heated to the boiling-point, then cooled to the ordinary temperature, diluted to 200 c.c. with the sodium chloride solution and filtered, and 100 c.c. of the filtrate titrated with the standard acid, using the same indicator. The difference between twice the number of c.c. consumed and that required by the 20 c.c. of sodium hydroxide solution corresponds to the amount of calcium and magnesium. By deducting the amount of magnesium previously determined and multiplying the difference by 1·36 the amount of calcium sulphate in grams per litre is obtained. C. A. M.

The Application of Bismuth Ammonium Molybdate to Gravimetric Analysis. Edmund H. Miller and Frederick Van Dyke Cruser. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 16.)—In this method the bismuth is precipitated as bismuth ammonium molybdate, as in Miller and Frank's method (*Journ. Amer. Chem. Soc.*, 1903, xxv., 926), and the precipitate ignited and weighed as $Bi_2O_3,4MoO_3$. To carry out a determination, a large excess (four or five times the theoretical quantity) of ammonium molybdate is added to the bismuth nitrate solution. A few drops of congo red are added, and then dilute ammonia until the solution becomes pink. A little dilute nitric acid is then added until the colour changes to lilac; the liquid is diluted to 150 or 200 c.c., and slowly heated, with occasional stirring. When the solution is at from 50° to 60° C., it is filtered through a platinum Gooch crucible, and the precipitate is washed with a 3 per cent. solution of ammonium nitrate until the volume of the filtrate is 400 to 500 c.c. The crucible with its cap on is next dried at 160° C.; when thoroughly dry it is moistened with a few drops of concentrated nitric acid, and heated at the tip of the flame of a Bunsen burner, so that the bottom is at not more than a dull red heat. Treatment with nitric acid and ignition are repeated until the precipitate is very light yellow in colour and the weight becomes constant. Results obtained by this method show a maximum error of

−0·0011 gram on about 0·3 gram bismuth. They are as good as those given by Miller and Frank's method (*loc. cit.*).

Incidentally it was found that in the method for the determination of bismuth by evaporation of the solution with nitric acid and subsequent ignition to oxide, porcelain, and not platinum, vessels must be used, as the latter cause low results owing to the passage of reducing gases through the red-hot vessel. A. G. L.

The Oxidation of Sulphites by Iodine in Alkaline Solutions. R. Harmann Ashley. (*Zeits. Anorg. Chem.*, 1905, xlv., 69.)—The author criticises Rupp's method (*Berichte*, xxxv., 3694), in which sulphur dioxide and sulphites are determined by allowing their solution to stand for fifteen minutes with an excess of iodine solution in the presence of 1 gram of sodium bicarbonate, and then determining the excess of iodine with thiosulphate. He finds that with moderate quantities of sulphur dioxide good results may be obtained by this method, which, however, are due to the balancing of two errors, one arising from the incomplete oxidation of the sulphite, the other from the fact that in alkaline solution a portion of the thiosulphate is oxidized further than to tetrathionate. With small amounts of sulphur dioxide the second error becomes very noticeable. A. G. L.

Influence of Sulphurous Acid on the Determination of Boric Acid. W. Vaubel and E. Bartelt. (*Chem. Zeit.*, 1905, xxix., 630, 631.) — Should sulphurous acid be present in a solution in which the boric acid is to be determined by titration in the presence of glycerol, using phenolphthalein as indicator, a preliminary treatment of the solution to remove the sulphurous acid is necessary; otherwise too low results are obtained. The removal of the sulphurous acid is best attained by boiling the solution after the addition of a strong mineral acid. To prevent loss of boric acid, a funnel or short reflux condensing tube should be placed in the neck of the flask in which the operation is carried out. Oxidation of the sulphurous acid by means of potassium permanganate, nitric acid, or hydrogen peroxide is not to be recommended. It might be mentioned that preservatives containing boric and sulphurous acids are now on the market. W. P. S.

Observations on the Use of Diphenylamine as a Reagent for Nitrites, Nitrates, and Chlorates. P. Alvarez. (*Bull. Soc. Chim.*, 1905, xxxiii., 717-719.) —Although nitrites invariably give the blue coloration with diphenylamine solutions, this is not the case with chlorates and nitrates. In practice the colours most frequently obtained are reddish-brown, changing to green, and finally gray with chlorates; and reddish-yellow, changing to green, and finally violet, with nitrates, the shades varying with the quantities of substances employed. The author therefore recommends a mixture of freshly prepared solutions in sulphuric acid of diphenylamine and resorcinol, or β-naphthol, as giving more permanent and distinctive tests. His reagent consists of 0·1 gram of crystalline diphenylamine and 0·1 gram of twice sublimed resorcinol in 10 c.c. of sulphuric acid (specific gravity 1·84). Five or six drops of this solution are added to 0·001 gram of the salt under examination in a small flat-bottomed crucible. *Nitrates* give a very stable yellowish-green colour, and on diluting the liquid and blowing over the surface the edges of the stain become blue. The addition of alcohol changes the colour to orange. *Nitrites* give an intense bluish-violet colour, and on shaking the crucible the edges of the stain appear red. The liquid itself becomes red on the addition of alcohol. In the case of chlorates this reagent does not give satisfactory results, but if the resorcinol be replaced by an equal weight of β-naphthol in the reagent, and the test applied in the same manner, a dull green coloration is obtained, which changes to dark gray—almost black. On the addition of alcohol the liquid remains gray or black. As these reagents are extremely sensitive, not more than 0·001 gram of the salts should be used in the test, especially in the case of chlorates and nitrites. C. A. M.

The Determination of Iodine Cyanide in the Presence of Iodine. J. Milbauer and R. Hac. (*Zeit. anal. Chem.*, 1905, xliv., 286-292.)—The mixture of the two substances is thoroughly rubbed with concentrated sulphuric acid in a mortar, and the contents of the mortar washed by means of sulphuric acid into a Kjeldahl flask, and diluted with an equal volume of water. The flask is now gently heated so that the iodine is expelled with the steam, after which the temperature is raised and the boiling continued for about an hour to convert the nitrogen into ammonium sulphate, and the ammonia distilled off and determined in the usual way. The results of the test experiments, described in detail, show that the method is extremely accurate. Nessler's reagent can be used for the detection of iodine cyanide or other nitrogenous compounds in the presence of iodine. The substance is first heated with dilute sulphuric acid (1 : 1), and the liquid after concentration to half its volume treated as in Kjeldahl's method. The distillate containing the ammonia in dilute sulphuric acid is concentrated to a small volume, and after being rendered slightly alkaline is tested directly with Nessler's reagent. All the samples of commercial iodine thus examined contained only insignificant traces of nitrogen. A solution of iodine cyanide in sulphuric acid is colourless, but becomes pink on standing, and on pouring water down the side of the tube a violet ring appears at the junction of the liquids. C. A. M.

Standardization of Permanganate Solutions by Means of Silver. K. Hopfgartner. (*Monatshefte f. Chem.*, 1905, xxvi., 469.)—The method used depends on the solution of silver by a ferric sulphate solution, the resulting ferrous sulphate being then titrated with the permanganate solution which is to be standardized. Since the reaction is reversible, a large excess of the ferric salt—preferably ferric ammonium sulphate—is used. To diminish the hydrolytic dissociation of the ferric salt, which, owing to the colour of the dissociated salt, interferes with the titration, the solution is made strongly acid with sulphuric acid. The form of silver found best was the finely-divided precipitate obtained by heating a solution of ferrous sulphate with silver sulphate. Quantities up to 2·5 grams of this silver may be dissolved in about twenty minutes at the boiling-point. Solution and titration are carried out in an atmosphere of carbon dioxide. Suitable proportions for a test are 0·7 gram silver and 25 grams ammonia alum dissolved in 150 c.c. of water and

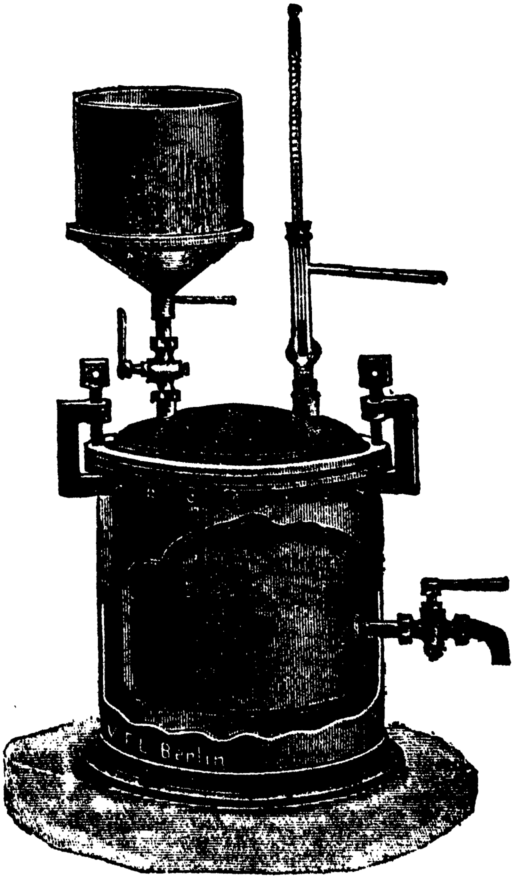
10 c.c. of sulphuric acid. When all the silver has dissolved, the liquid is cooled and diluted to about 600 c.c. with 10 per cent. (by volume) sulphuric acid. Two solutions, the iron values of which had been found to be 3·0222 and 28·300 grams respectively per litre by the hydrogen peroxide method, were found to have the iron values 3·0249 and 28·255 respectively.

Since 1 gram of silver corresponds to 0·51773 gram of iron, and 1 gram of copper corresponds to 1·7573 grams of iron, silver-copper alloys may be analysed by dissolving a weighed amount in ferric sulphate solution and titrating with permanganate; but solution takes a considerable time in this case. Thus, in a silver-copper alloy containing 83·50 per cent. of silver, 83·57 per cent. of silver were found by this method.

A. G. L.

APPARATUS.

Apparatus for Evaluation of Coal-tar. A. Rispler. (*Chem. Zeit.*, 1905, xxix., 488.)—The apparatus illustrated by the accompanying figure has been designed by the author to avoid the difficulty usually experienced from the boiling over of the tar on distillation.

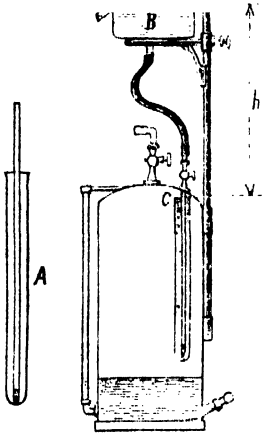


The apparatus consists of a still which is capable of holding 5 or 6 kilograms of tar. The top of the still has two short tubes, one of which is fitted with a glass T-piece carrying a thermometer and connected with a Liebig's condenser; the other connected through a tap to a funnel-shaped vessel, which can contain about 1½ kilograms of tar. There is another tap near the bottom of the still, and the still itself is heated by a large burner.

For use, a quantity of 5 to 6 kilograms of tar is weighed out, and of this about 1½ kilograms poured into the funnel-shaped vessel; about half this quantity being let into the still. If the tar is very viscous, the funnel is heated by hot water or steam. The still is then heated and the tar begins to distil. When the thermometer reaches 110 to 120° C., the tap from the funnel is opened and tar run in at a rate that allows the temperature gradually to rise to 130°, which is then maintained throughout the operation by regulating the current of the incoming tar. In this manner the whole 5 to 6 kilograms can be distilled within an hour, the funnel being, of course, filled up as required. This estimation of water and light oil being completed, any further determination can be carried out in the ordinary manner. The apparatus lends itself to a number of successive determinations; the tar deprived of water and light oil can be run off through the bottom tap, and the apparatus is then at once ready for a second experiment. The author states that five or six such determinations can be made in this manner in one day. The apparatus can be obtained from the firm "Vereinigte Fabriken für Laboratoriumsbedarf," Berlin, N.

E. K. H.

Gas-Holder with Constant Flow. **Mario Betti.**



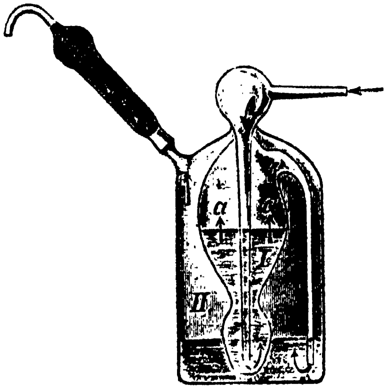
(*Chem. Zeit.*, 1905, xxix., 219.)—An improved form of Mitscherlich's gasometer in which, by suitable construction of the water inlet-tube, a flow of gas at a constant pressure is obtained.

Instead of the usual inlet-tube reaching to the bottom of the container, a double concentric tube is employed, of the shape shown in the figure. This forms a hydraulic valve, which allows water to flow into the container until the pressure of gas in this is equal to the weight of the column of water above the vessel (*h*). By varying the height of the upper reservoir, and maintaining the level of the water in this, the gas pressure may be kept at any desired point.

H. A. T.

Potash Apparatus for the Rapid Absorption of Carbon Dioxide. **G. Schöler.**

(*Chem. Zeit.*, 1905, xxix., 569, 570.)—The form of the apparatus is shown in the accompanying illustration. In the inner part of the apparatus (*I*) is placed a concentrated potassium hydroxide solution containing a little soap. As the gases bubble through the solution, a froth is formed which greatly aids absorption of the carbon dioxide. The outer part (*II*) contains concentrated potassium hydroxide solution, to which has been added a small quantity of calcium chloride, or the solution may be made up with hard water. This destroys any of the froth which is carried over into this part of the apparatus, and prevents fouling of the calcium chloride drying-tube. The soap solution is prepared by dissolving 5 grams of ordinary yellow soap and 50 grams of potassium hydroxide in 150 c.c. of water The solution is warmed and filtered. For the second solution 100 grams of potassium hydroxide and 0·04 gram of calcium chloride are dissolved



in 100 c.c. of water. Filtration is not necessary. The combustion gases may be passed through the apparatus at the rate of ten bubbles per second. W. P. S.

Apparatus for Determining the Melting-point of Organic Substances.

A. Landsiedl. (*Oesterr. Chem. Zeit.*, 1905, viii., 276, 277.)—The general form of the apparatus and its various parts are shown in the accompanying figures. A thermometer (*T*, Fig. 1) and a capillary tube (*r*) are placed in a tube (*a*) 25 cm. long and 15 mm. wide, closed at the bottom and provided with a cork at the top, through which the capillary tube and thermometer pass. This tube (*a*) is placed in the wider tube (*b*), which is two-thirds filled with sulphuric acid. A small side-tube (Fig. 2) or hole (*e*, Fig. 3) prevents pressure being formed in the acid vessel (*b*). The latter stands on a stout piece of wire-gauze, and may be surrounded by a glass jacket (*c*), placed on an asbestos ring (*d*). The substance

FIG. 2. FIG. 1. FIG. 3.

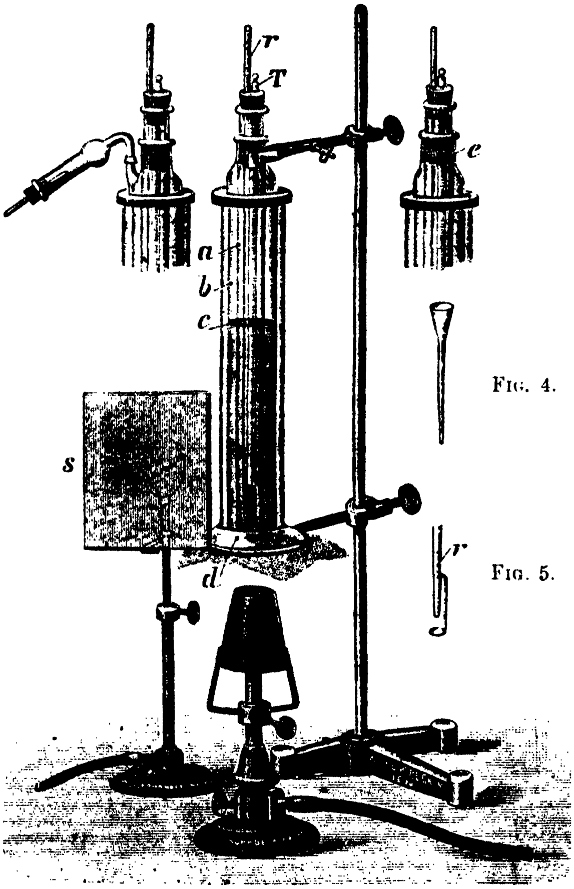


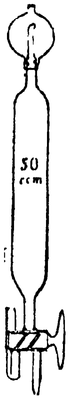
FIG. 4.

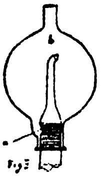
FIG. 5.

of which the melting-point is to be determined is placed in a capillary tube, shaped as in Fig. 4. The widened top of this is held by the slightly constricted bottom of the capillary tube (r), so that the substance is brought close to the bulb of the thermometer. If ordinary cylindrical capillary tubes be used, a small cup-shaped support is placed on the tube (r, Fig. 5) to hold them in position. A screen (s) before the illuminating flame will be found useful. The apparatus gives a very steady rise in temperature. In determining a melting-point, it is recommended that a preliminary experiment be made, and the actual melting-point then taken, commencing with the temperature a few degrees below the point thus roughly obtained.

W. P. S.

New Automatic Pipette. Von Greiner and Friedrichs. (*Zeits. f. angew, Chem.*, 1905, xviii., 465.)—In order to avoid contamination with rubber—which is a defect in the earlier automatic pipette brought out by one of the authors some twenty years ago—this new instrument is fitted with a closed cap, the neck of which is provided with a channel corresponding to an opening in the pipette. By rotating this cap the overflow of liquid can be easily let into the pipette again (Figs. 1 and 2).





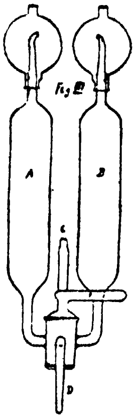
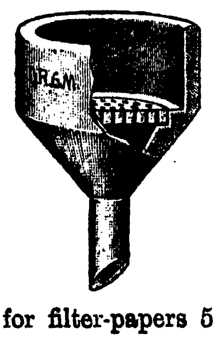


Fig. 3 shows a double automatic pipette, the tap of which is so arranged that one pipette is filled from the reservoir whilst the other is being emptied. Obviously the reservoir must also be provided with a tap by which the inflow can be regulated.

E. K. H.

Improved Suction Filter. J. Katz. (*Chem. Zeit.*, 1905, xxix., 489.)—As seen from the figure, the perforated plate, instead of being a fixture and occupying the whole width of the cylinder, is removable, and from 2 to 3 millimetres less in diameter, a rebate being provided to receive it, the height of which corresponds with the thickness of the plate. By this arrangement the filter-paper lies perfectly flat, and no wrinkles are formed at its edges through which a portion of the precipitate may escape. The funnel is readily taken in pieces to clean. A porcelain ring is also supplied, which may be placed inside the funnel to hold down the filter-paper, when it is necessary to pour off the liquid from above the precipitate. The funnel is manufactured in three sizes, for filter-papers 5½, 9, and 15 cm. in diameter respectively, by F. Hungershoff, of Leipsic.



E. K. H.

The Ashe-Finlayson Comparascope.—This ingenious addition to the microscope may at times prove of considerable value to the analyst, for it enables him to visually place two objects in the same field of view, thus enabling an exact comparison to be made between the two. As shown in the accompanying figures, the apparatus is attached to the nose of the microscope by an adaptor (*A*), which takes two objectives, one being at a right angle with the other. To a horizontal arm fixed to the adaptor a fine-focussing stage, with clips (*SS'*), is provided, also a mirror (*M*), and for high powers a condenser (*C*) with iris diaphragm (*I*). The rays from the vertical objective and those from the horizontal one are divided into two series by a prism (*P*), so that in half the field the objects on the slide at *S* are seen, in the other half those on the ordinary table of the microscope. A division (*E*) carried a short way up the tube serves to prevent confusion in the two half fields. The prism (*P*) can be turned out of position by the milled head (*L*), thus throwing the comparascope out of use when the instrument acts as an ordinary microscope. The apparatus is manufactured by Messrs. R. and J. Beck, of London.

W. J. S.

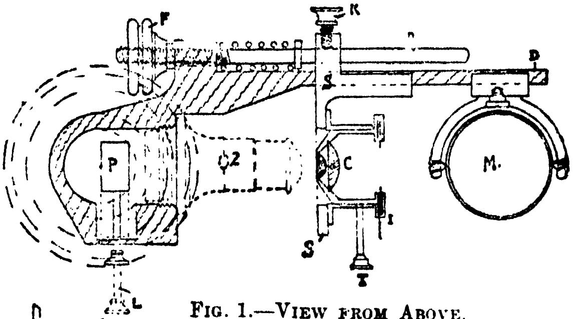


FIG. 1.—VIEW FROM ABOVE.

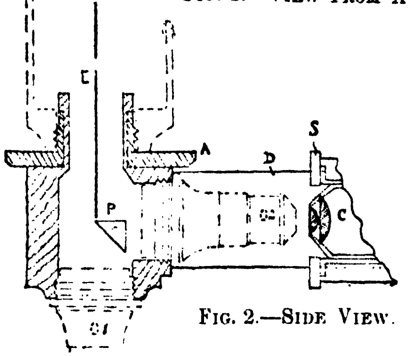


FIG. 2.—SIDE VIEW.

Electrically Heated Carbon Tube Furnaces. R. S. Hutton and W. H. Patterson. (*Chem. News*, 1905, xci., 272, 285.)—The authors describe several forms of electrically heated furnaces, in all of which the heat is obtained by passing a current through a carbon tube. These carbon tubes may be bored from graphite; in this case the current is led in through copper connections fastened to graphite plates into which the ends of the carbon tube are screwed. It is better, however, to use tubes of agglomerated carbon, which may be obtained from the makers of arc carbons. With these tubes connection is made by roughening the ends somewhat, plating them with copper, and then soft-soldering them to thick copper tubes just large enough to slip over them. The copper tube is water-jacketed for a short distance to protect the joint; at its other end a copper clamp connects it to the flexible cable. Joints made in this way last exceedingly well.

To prevent the carbon tubes from burning and to keep in the heat they are surrounded with magnesia, or, preferably, powdered carborundum, kept in place by suitably arranged brickwork. The carbon tubes are also extended by glass tubes inserted in the ends by means of asbestos string; through the glass tubes hydrogen or other gas may be led through the carbon tube. A glass T-tube may conveniently be used at one end of the carbon tube to furnish a spy-hole through which the interior of the tube may be observed. In order more thoroughly to exclude carbon monoxide and other oxygen gases, it is advisable to pass hydrogen outside as well as inside the carbon tube. This may be done by leading the gas into the whole space occupied by the carborundum. A better method consists in surrounding the carbon tube through which the current is passing with another carbon tube, also soldered to thick copper tubes at the ends. This outer carbon tube is surrounded with carborundum; the space between the two carbon tubes is filled with hydrogen.

As regards dimensions and current required, a graphite tube 28 cm. long, 2 cm. external and 1·5 cm. internal diameter jacketed with carborundum, using a current of 320 ampères at 9·6 volts, melted nickel in thirteen and platinum in sixteen and a half minutes; an agglomerated carbon tube, 27 cm. long, 2 cm. external and 1·5 internal diameter, jacketed with carborundum, using a current of 140 ampères at 7·7 volts, melted nickel in nineteen, and platinum in twenty-eight minutes; a larger agglomerated tube, 60 cm. long, 8·2 cm. external and 6·7 cm. internal diameter, using 600 ampères at 8·6 volts, gave a temperature of 1200° C. in thirty minutes; with 850 ampères at 13·0 volts it melted nickel in twelve and platinum in twenty and a half minutes.

As may be seen, all these furnaces are designed to work with high-current densities at low pressures. It will probably be possible to adapt them to high voltages by cutting a spiral in the tube, thus leaving a helix of much higher resistance (*Eng. Pat.*, 24,472, November 18, 1903). A. G. L.

* * * * *

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST OF THE JULY EXAMINATIONS.

Of nineteen candidates who entered for the Intermediate Examination, twelve passed: J. W. Agnew, H. G. Dale, James Dick, jun., W. P. Hayworth, I. M. Heilbron, R. F. Innes, S. Judd Lewis, A. J. C. Lickorish, F. L. Okell, R. P. Page, H. J. B. Rawlins, B.Sc. (Lond.), and D. R. Wood. In the Final Examination for the Associateship (A.I.C.), of five examined in the branch of Mineral Chemistry, three passed: J. B. Hoblyn, Assoc.R.C.Sc. (Lond.), G. W. James, B.A. (Oxon.), and H. H. Kingdon, B.A. (Oxon.); of six in Organic Chemistry, four passed: A. W. Bain, B.A., B.Sc. (Lond.), T. W. Cheke, J. Stuart Hills, and D. F. Twiss, M.Sc. (Birm.); and of nine who entered in the branch of the Analysis of Food and Drugs and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following four passed: R. W. Blair, Assoc.R.C.Sc.,I., P. H. Carpenter, J. A. Goodson, and S. Summerson, B.Sc. (Lond.). One candidate passed an examination for the Fellowship (F.I.C.): E. F. Harrison, B.Sc. (Lond.). The examiners in Chemistry were Mr. W. W. Fisher, M.A., F.I.C., of Oxford, and Dr. G. G. Henderson, M.A., F.I.C., of Glasgow. The examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., M.B., F.R.S., F.I.C., of Cambridge.

SEPTEMBER, 1905. Vol. XXX., No. 354.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTES ON THE HISTORY OF DISTILLED SPIRITS, ESPECIALLY WHISKY AND BRANDY.

By T. FAIRLEY, F.R.S.E., F.I.C.

(*Read at the Meeting*, *May* 3, 1905.)

Beverages, liquids obtained by fermentation of the juices of fruits or other parts of plants, have been in use all over the world from the earliest times. It is even said that there is evidence of the use of malted barley long back in the prehistoric period, during the Stone Age, and in the lake dwellings of Switzerland, etc.

Failing saccharine juices from plants, the milk of animals has been used for this purpose, as shown in the koumiss of the Tartars in Central Asia and the kephir of the inhabitants of the Caucasus.

It is not so generally accepted that the art of distilling fermented liquids to obtain a more concentrated alcoholic liquid is also of unknown antiquity. In many works on the chemical history of Modern Europe the Arabians are credited, not only with having conveyed the knowledge of distillation to Southern Europe, but also that Geber (about A.D. 800) invented the process. Certainly Rhases, one of Geber's followers, used the process in the distillation of fermented materials; and about a century later Albucasis, a physician of Cordova, gave an exact description of a distillation apparatus as applied in the preparation of medicines, among which l'eau de vie de vin was reckoned one of the most valuable. For some centuries this was the chief application of the spirit from wine, and the knowledge was spread into France and other countries by Arnaud de Villeneuve, Raymond Lully, and others who lived at that time.

In ancient times such processes were often kept secret* in the hands of the priests or religious houses, and later wines and spirits specially flavoured were produced by these houses, some of which survive to the present day.

Distillation is seldom referred to by Greek or Roman writers, though Aristotle in his "Meteorology" says: "Sea-water can be rendered potable by distillation; wine and other liquids can be submitted to the same process. After they have been converted into humid vapours they return to liquids."†

Aristotle also explains the formation of dew by the condensation of the vapour of water suspended in the air.

If these views of Aristotle were ever applied in practice it was done secretly, and the first exact description of apparatus for distillation in this part of the world is by Zosemus of Alexandria (about the fourth century). He quotes the figures as taken from the ancient temple of Memphis in Egypt.‡

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* Kept secret under the most severe penalties, Hoeffer's "Histoire de Chimie," vol. i., p. 33.

† Aristotle's "Meteorology," lib. ii., chap. ii.; Hoeffer's "Histoire de Chimie," vol. i., p. 98.

‡ Hoeffer's "Histoire de Chimie," vol. i., p. 262.

But not merely in Egypt, but over a great part of the Eastern world, the knowledge of distillation prevailed from times of an unknown antiquity.

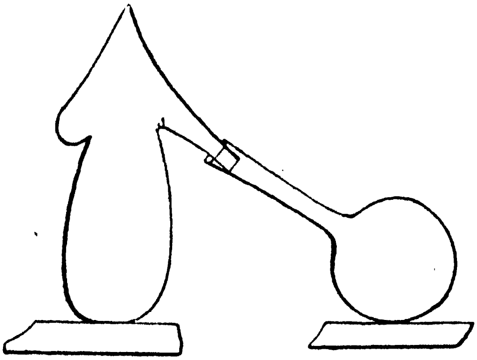
I append a table showing some of the alcoholic beverages obtained by fermentation and the distillates obtained from them. In India and some other countries a number of different materials were used for these purposes, as they are to this day. Only the more important ones are shown on the table :

TABLE OF FERMENTED AND DISTILLED LIQUIDS USED IN VARIOUS COUNTRIES.

Country.	Raw Material.	Fermented Liquor.	Distilled Liquor.	Period of Discovery.
China.	Rice, millet, etc.	Tar-asun or tchoo.	Sautchoo.	Long before Christian era.
Japan.	Rice.	Saké.	Sochu.	Very ancient.
India.	Rice and other grains. Flower spathes of various palms. Flowers of Muowha-tree.	Toddy and other names.	Arrack.	At least 800 B.C.
Ceylon.	Palm-tree (*Cocis nucifera*).	Toddy.	Arrack.	"Time immemorial."
Thibet.	Rice, wheat, etc.	Chong.	Arra.	Ancient.
Tartary.	Milk of mares, etc.	Koumiss.	Arika and other names.	Very ancient.
Caucasus.	Milk of mares, etc.	Kephir.	Skhou and other names.	
Peru.	Maize and manioc.	Chica, sora.	Puichiu.	Unknown, but in use soon after Spanish Conquest.
Mexico.	Agave.	Pulque, octli.	Mexical, agua ardiente de maguey.	
South Sea Islands.	Ti or Tee root.	—	Ava.	?
Continent of Europe and Egypt.	Barley or other grains.	Beer and other names.	?	?
	Honey.	Mead, etc.	?	?
	Grape.	Wine.	Eau de vie de vin, etc.	Ninth century.
British Islands.	Barley.	Beer.	Uisque beatha, whisky.	Before twelfth century.
	Honey, etc.	Mead.	"Mead distilled."	Ancient British.

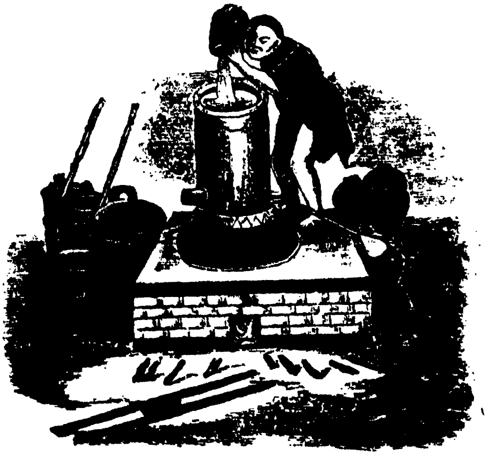
AUTHORITIES QUOTED IN TABLE: "Encyclopædia Britannica," and Supplement, ninth edition, vol. ii., p. 628; vol. iv., p. 216; vol. vii., p. 264; vol. xiii., p. 263; vol. xvi., pp. 213, 305; vol. xxxii., p. 806; articles by Dittmar, Paton, Schidrowitz, etc. Cyrus Redding's "History of Modern Wines," Appendix XXIX., p. 836; Morewood's "Inebriating Liquors," second edition; Muspratt's "Chemistry"; Scarisbrick's "Spirit Manual," 1884; Scarisbrick's "Spirit Assaying"; Valpy French's "Nineteen Centuries of Drink in England"; Hoeffer's "Histoire de Chimie," 1866; Skeen's "Wines and Other Fermented Liquors"; Tovey's "Wine and Wine Countries."

The following illustrations show some of the ancient forms of stills.



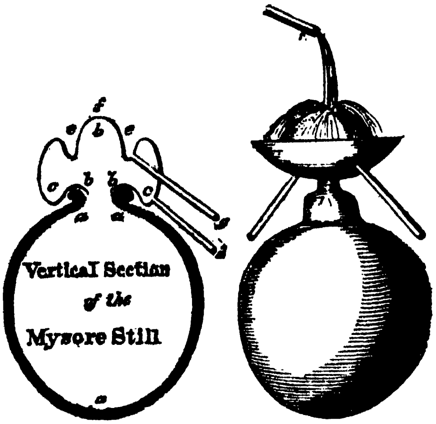
Alembic and Receiver figured by Zosemus the Panopolitain, of Alexandria, said to have been copied from the Ancient Temple of Memphis, in Egypt.

He wrote in Greek, and a copy of part of his writing is preserved in the "Bibliothèque Imperiale" in Paris, and is quoted and transcribed by F. Hoeffer in his "Histoire de Chimie," vol. i., p. 252 and appendix. In this connection it is interesting to note that the word "alembic" is really a Greek word αμβιξ with the Arabian article "al" prefixed. Our word "beaker" may also be derived from αμβιξ, gen. αμβικος, a cup, etc., from βικος.



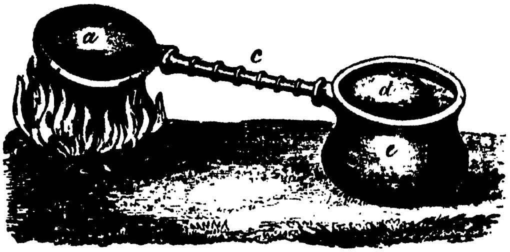
Still used in China.

This is probably one of the earliest forms of still without the worm-tub. The head of the still is generally globular, and is enclosed in the lower part of the cylinder kept filled with cold water. The neck from the head of the still projects outside the cylinder, whence a small gutter or tube conveys the condensed liquid to the receiver.—From Du Halde's "Description géographique, historique et physique de l'empire de la Chine et de la Tartarie Chinoise," vol. i., p. 303; and in Morewood's "Inebriating Liquors," 1838 edition, p. 228. See also Dr. H. Gray's "China," p. 141.



Ancient Still used in Central India.

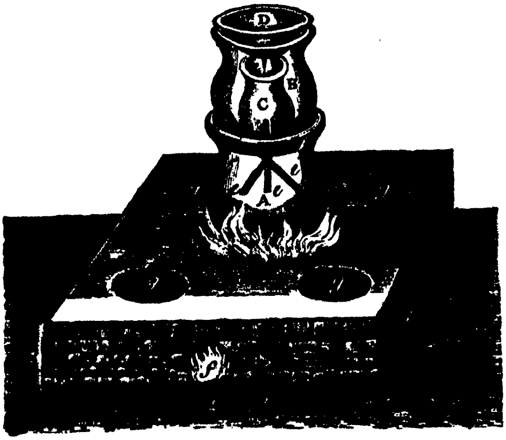
In use, a hole is dug in the ground suitable for the size of the body of the still, with room for the fire below, and level with the bottom there is underground communication with a second excavation for the purpose of feeding the fire. A fire is first kindled in the pit, and the ground thoroughly heated (as in the ancient mode of baking bread in the East). A chimney is also provided near the edge of the hole. Then the still is charged with the fermented liquid from rice, molasses, the juice of the cocoanut-tree, etc., and fixed in the pit and enclosed with earth round the top of the still, so that no heat can escape. On boiling, the vapour is condensed by a stream of cold water constantly applied by the attendant, running off at *g*, while the condensed liquid runs off at *a*. These stills may be of metal or earthenware.—Buchanan's "Journey through the Mysore," vol. i., p. 39; Morewood's "Inebriating Liquors," p. 159.



Ancient Still used in Ceylon of the Simplest Construction, and still in use by the Natives.

a., *b.*, is the alembic and capital luted together; *d.*, *e.*, a refrigerator and receiver of one piece, and the latter connected with the head by a bamboo, *c.*

The still employed for this purpose is of earthenware and of the simplest construction. The above is a true representation of the one in general use.—Morewood's "Inebriating Liquors," p. 186; Muspratt's "Chemistry," vol. i., p. 115.

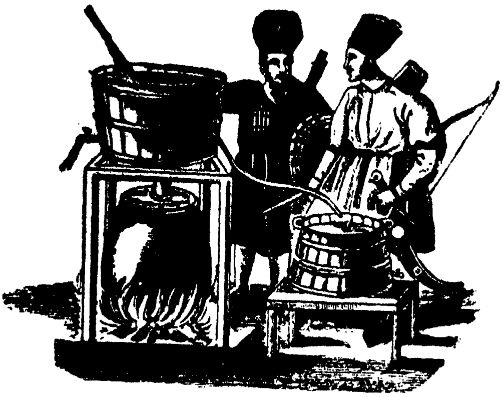


STILL USED IN THIBET AND BOOTAN.

A, An earthen vessel, in which the chong is placed, immediately over the fire; *B*, another vessel without a bottom; *C*, a smaller earthen vessel, which is the recipient; *D*, an iron basin filled with cold water, renewed occasionally as it grows warm, which may be termed the condenser; *e*, *e*, *e*, the three cross staves of wood on which the recipient is placed. The junction of the three vessels (*A*, *B*, and *D*) being secured with cotton bandages and clay lute, a fire is lighted under *A*, which contains the chong. The spirit rises through *B*, is condensed upon the convex bottom of the basin (*D*), and is received into the smaller vessel (*C*). *f*, The fireplace; *g*, *g*, *g*, openings over the fire for the reception of similar apparatus.—Turner's "Embassy" (1800), pp. 26 and 348; Morewood's "Inebriating Liquors," p. 172.

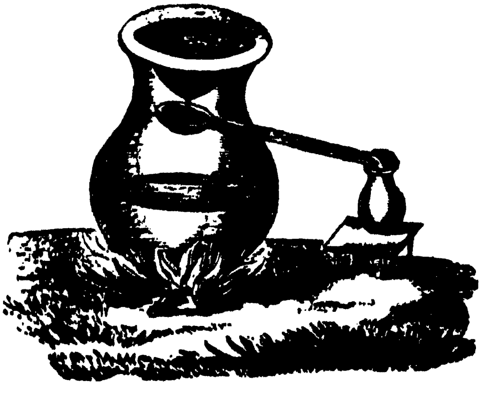
ANCIENT STILL USED BY THE KALMUCK TARTARS, CONSTRUCTED OF COARSE CLAY, CONNECTED WITH THE RECEIVER BY A TUBE OF CANE OR WOOD.

The head of the still is kept cool by coating with wet mud and the application of cold water. The koumiss gives at first a weak spirit, which may be further concentrated by a second or third distillation. They have separate names for these distillates, according to the number of times the liquid has been distilled.—Clarke's 'Travels in Asia," etc.; Morewood's "Inebriating Liquors," p. 147.



STILL USED BY THE TRIBES OF THE CAUCASUS.

The pot is made of earthenware or of copper, covered with a lid luted on, and carrying a tube connected with the condenser overhead. This apparatus gives a nearer approach to the worm-tub than any other primitive form of still.—Morewood's "Inebriating Liquors," p. 507.

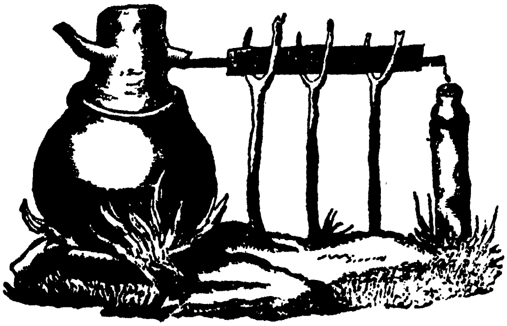


ANCIENT FORM OF STILL USED BY THE PERUVIANS.

The fermented liquor to be distilled is put into a deep earthen pot having a hole in the side near the top, through which passes a wooden gutter of the form shown, connecting with the receiver. Over the top a pan filled with cold water and luted to the pot with clay is placed. This acts as the condenser, and the spirit flows along the groove into the bottle or receiver.—Morewood's "Inebriating Liquors" (1838), pp. 138 and 306.

An apparatus similar in arrangement but generally made of copper is in use in Japan.—Roscoe and Schoelemmer's "Chemistry," vol. iii., part i., p. 296.

It is probable that the Peruvians used this apparatus long before the date of the Spanish Conquest.



Primitive Still used in Tahiti, the Largest of the Georgian Islands.

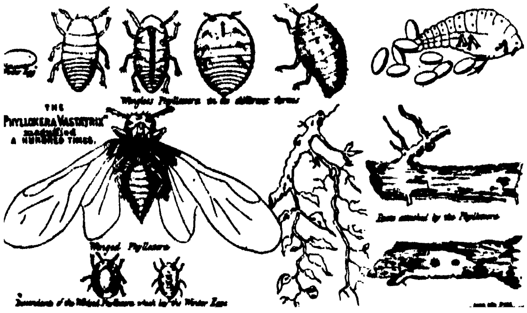
In many of the South Sea Islands the knowledge of distillation and of distilled liquors has been introduced by Europeans. But Captain Cook, when he first visited the Georgian Islands, found them already in possession of this knowledge, with an apparatus which, from its most primitive form and materials, may have been known for many centuries.

The body of the still is a large stone hollowed to form a pot, placed on stones with spaces between for the fire. To the top of this pot is placed a head formed of the hollowed-out trunk of a tree. In this is inserted a long bamboo cane, which passes through a trough or gutter filled with cold water to serve as a condenser.—Ellis's "Polynesian Researches," vol. i., p. 230; "Encyclopædia Britannica," ninth edition, vol. vii., p. 264; Morewood's "Inebriating Liquors" (1838), p. 256.

Illicit Still as used in Ireland (Eighteenth Century).

Up to 1556 the distillation of spirits was carried on in Ireland without license or taxation. The attempts to raise revenue by granting monopolies or licenses, and, later, by taxation on the liquids produced, were long opposed and resented by the people, and the illegal manufacture often exceeded the amounts on which taxes were paid.

The apparatus was of the simplest kind. The common black pot of the kitchen supplied the body of the still, to which were attached simple fittings such as could be replaced in case of seizure a a small cost. Failing an authentic drawing of a still used by the natives centuries ago, I give from Morewood, p. 675, a view of an Irish illicit distillery. Watchers are posted on the hills, and the police or revenue officers are to be seen approaching on the left.


THE
PHYLLOXERA VASTATRIX"
magnified
A HUNDRED TIMES.
Wingless Phylloxera in its different forms
Winged Phylloxera
Roots attacked by the Phylloxera
Descendants of the Winged Phylloxera which lay the Winter Eggs

PHYLLOXERA VASTATRIX.

The great crisis which arose about twenty years ago in France and other wine-growing countries was due to the ravages of a minute insect figured above. The destruction was very great, and in the Charente district the vines were almost exterminated by the exceeding voracity and enormous reproductive powers of the species. The winter egg (magnified) is seen in the left top corner, and to the right of this egg the wingless (aptera) insects developed from it in the spring, mostly females. Each of these produces an enormous number of eggs, hatching again, mainly to females, and so on for eight to ten generations during the summer. In August or September the winged insect appears—male and female—living only a short life, but easily transported by the wind to settle on the leaves and lay three or four eggs under the bark. The peculiar sexual arrangement common to certain species of only requiring fertilization from the male once in so many generations facilitates rapid multiplication (parthenogenesis, meaning "virgin birth"). The insects feed mainly on the roots of the vine, and the wounds they make cause loss of sap and rapid decay. In view of the great importance of the subject, I have ventured to add this illustration and description, from Tovey's "Wines and Spirits," p. 284.

In each case the fermentable material most convenient or available in the country is used for the purpose of fermentation and distillation. There is no evidence that there ever was a period when all alcoholic spirit was obtained from wine, as applied generally over the world, and certainly not as applied to the British Islands.

The British, like other Northern nations, made a fermented liquor from honey (mead and other names), and in the "Mead Song," written in the sixth century by Taliesin, a British poet, the words occur: "Mead distilled I praise, its eulogy is everywhere,"* etc. I have not found any other reference to distilled mead.

WHISKY.

This spirit was probably first prepared in the East. Barley was especially fermented in those countries subject to inundation, such as the greater part of fertile Egypt, where the vine does not flourish. In India the fermentation of barley was forbidden in times of scarcity or famine. They distilled these fermented liquors from rice and other grains from a period before the Christian era.

This knowledge gradually came to the Arabians, and Rhases, in Hoeffer's "Histoire de Chimie" (vol. i., p. 342), describes a process for preparation of eau-de-vie by a simple process from "occulti," or secret thing, which Hoeffer explains to have been wheat allowed to germinate in the ground.

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* Sharon Turner's "Vindication of the Ancient British Poets," 1803; Valpy French, "Nineteen Centuries of Drink in England," p. 15.

When the knowledge of distillation first came into the British Islands is uncertain, but such knowledge was independent of the distillation of a spirit from wine, seeing that the process was in use in Ireland before the latter came into Europe.

The Irish legend states that St. Patrick taught the art of distillation as applied to "potheen," which is said to take its name from that of the saint.*

Whether or not the date of the distillation of whisky be as old as this legend would make it, it is certain that when the English first invaded Ireland, 1170-1172, the manufacture of whisky was a matter of common knowledge among the people of the country. Like the process of brewing, it was carried on as a domestic art, and was not subject to any legal restrictions till some centuries later. As on the Continent, the Irish distilled liquor was used largely as a medicinal agent, and its Celtic name, "uisque beatha," English "usquebaugh," means "water of life." This name is found in the old writings and songs of the country. By dropping the last syllable, usquebaugh has been transformed into the modern word "whisky."

The various Esks—names of rivers in England and Scotland—and the Usk in South Wales, are forms of the same word—meaning water.

In the early times of the Christian era Ireland was a more civilized country than a great part of Europe, and there is proof that the Irish had direct intercourse with the East. The Phœnicians traded with Cornwall and the adjacent parts of Ireland, and in all probability the knowledge of distillation came direct into Ireland from the East. The modern word "Fenian" expresses the claim of the Irish to be related to the ancient "Phœnicians."

In the time of Henry VIII., Irish settlers came over into Pembroke and established distilleries,† and no doubt the soldiers of Henry II., 300 years before, brought back with them the knowledge of whisky.

The population in the islands, etc., of the South-West of Scotland has been largely recruited from Ireland, and distilling has been carried on there probably almost as early as in Ireland itself. In the time of the Tudor Kings Scotch whisky attained considerable reputation in England.

Legal documents referring to aqua vitæ made from malt in 1494‡ are extant, and the manufacture was common at that time, and had been carried on long before as a domestic art, and in the religious houses. Most large houses had their stillroom—"stillatory." From the time that the manufacture of spirits was made a source of revenue either by licenses to use stills (Tudor and Stuart period) or by excise duties on the amount manufactured (middle of seventeenth century and onwards), we have no difficulty in tracing the history of distilled spirits. The dispersion of the religious houses at the time of the Reformation helped to diffuse more generally the knowledge of the arts practised by the monks—among them that of distillation.

In Scotland and in Ireland in former times, whisky was prepared from malted barley, and the genuine *small-still* whisky in Scotland is still prepared in this way.

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* Chambers' "Book of Days," vol. i., p. 384, and vol. ii., p. 64.

† Valpy French, "Nineteen Centuries of Drink in England," p. 133.

‡ "Accounts of the Lord High Treasurer of Scotland," edited by Thomas Dickson, 1877, vol. i., p. ccxiii.

In Ireland the malted barley is mixed with a certain proportion of unmalted barley or other grain, though there are distilleries in Ireland which use malt only.*

In modern times the manufacture of spirit has obtained an immense development in the patent stills so largely used. These give a spirit containing much less of the other substances which go over with the spirit in the small stills, and to which, after ageing, the special flavour of the small-still whisky is due. In certain parts of England this plain patent-still spirit, after diluting and colouring, is sold as whisky, but much of it is used for blending with the small-still whisky, which costs more to produce, and some of the best of these blends are only usable after ageing for a certain number of years.

Like the best brandies, some of the best whiskies are scarcely usable when new, but when kept for a sufficient time their character changes greatly. So far as my observation goes, the genuine small-still whisky, properly aged, is less injurious than one would expect an alcoholic liquid to be of the same strength.

The "blending" of spirits may save a certain time in the ageing, when its value is locked up for so many years; it also saves the difference in cost between the small-still whisky and the patent-still spirit. If the patent-still spirit used be obtained from grain the evil of mixing is less than if it is obtained from potatoes, which gives a spirit more difficult to purify.

Let us now trace the variations in the trade meaning of "whisky."

1. The small-still distillate from malt only.
2. The small-still distillate from a mixture of malted barley and some other grain.
3. In the United States and Canada the spirit distilled from fermented maize, rye, etc., often, therefore, called corn whisky.
4. Blended mixtures of small-still malt whisky and patent-still spirit, the latter generally obtained from grain.
5. Small-still spirit with patent-still spirit from potatoes, molasses, beetroot, etc.
6. Patent-still spirits, slightly coloured, flavoured or unflavoured.

The consumer ought to have the right to obtain either the pure malt small-still whisky or any of the others mentioned above to suit his requirements.

The selling of a liquid which costs less in the name of one which costs more must ultimately lead, when it becomes known and understood, to a diminution in the demand and sale.

BRANDY.

Early in the fourteenth century Genoese traders brought arrack from the East into England and this is the first known importation of foreign spirits by sea. In Queen Elizabeth's time the trade in brand-wine from France had attained considerable dimensions. The trade was in the hands of the French, English, and Dutch. The distillation of brandy began to take form in France as a manufacturing industry early in the fourteenth century. Some authors give 1313, others 1343. It became subject to legal restriction and control about 1619 in France and in 1590 in Spain.[*] The extension of the term "brandy" to include distillates not made from wine, though flavoured and coloured to imitate the genuine wine distillate, practically began in France, and received enormous development during the latter half of last century.

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* See Allen on "Whisky," *J. S. C. I.*, 1891, p. 305.

The modern English word "brandy" is quite as old as any of its Continental equivalents. As shown by the following examples, it was formerly spelt brandewine, brand-wine, and brandy wine.

The word "brand" is common to all the Teutonic languages of Northern Europe,[†] and means a thing burning, or that has been burnt, so that brandy or brandy wine, meaning burnt wine, is as old as the High German branntwein, the Dutch brandewijn, or the old French brandevin.

In Spain and other countries, when wine goes off in quality so as not to be saleable, it is sent to be "burnt"—that is distilled.

Examples of the meaning and application of "brand":

English Bible: "A brand plucked out of the fire."

Modern English: "Brandnew," originally applied to castings, forgings, etc., fresh from the fire. "To brand," to mark or burn with a hot iron. "Brand," the mark made by a hot iron, or the hot iron itself, etc. "Brand," a flashing sword, is from the same root, through the Romaic and Italian "brando," whence our word "brandish," and the French word "brandon," a torch, etc.

Examples from Murray's "Etymological Dictionary" and other sources showing the changes in the word "brandy."

Brandwine[‡] *and Brandewine.*—1622, Fletcher's "Beggar's Bush": "Buy brand wine." 1650, Roxburgh "Ballads": "It is more fine than brandewine."

Brandywine.—1652, Proc. Parl., No. 132, 2,391: "Laden in wools, brandywine, and salt." 1697, View Penal Laws, 173: "No aqua vitæ or brandywine shall be imported into England." 1729, D'Urfey's "Pills," etc., v. 23: "I was entertained with kisses fine and brandywine."

Brandy.—In familiar use this word came in about 1657 by dropping the last syllable from brandywine, but the latter was retained in legal documents down to 1702.

The best early description which I have been able to find of the distillation of wine is given by Ortholain, who lived in Paris about the middle of the fourteenth century. He published in 1358 "Practica vera Alchemica per magistrum Ortholanim, Parisiis probata et experta, sub anno Dom. 1358. Theatr. Chem., 4 p., 1028." Hoeffer[§] quotes from him and says:

"In a remarkable chapter on the distillation of wine he describes the manufacture of eau de vie de vin of different degrees of concentration, and he indicates how to obtain absolute spirit of wine. The process which he describes is not greatly dissimilar to that in use in the small-still distilleries at the present day."

Brandy, up to a comparatively modern period, was the spirit obtained from wine and from wine only. During the last fifty years the manufacture of factitious brandy has attained an enormous development both in France and in other countries. I quote from official sources statistics of the brandy trade in France from 1840 to 1900. It is seen that " trade brandy," which in 1850 formed 10 per cent. or less of the total production, in 1893 formed 90 per cent. or over of the whole production.

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[*] Morewood, "Inebriating Liquors," p. 366.

[†] Kluge's "Etymological German Dictionary."

[‡] Shakespeare apparently thought these words insufficient: *Othello*, Act II., Scene 3: "O thou invisible spirit of wine, if thou hast no name to be known by, let us call thee devil."

[§] "Histoire de Chimie," 441-443.

The brandy exported into the British Islands as pure eau de vie de vin from France, exceeds the total production of brandy from wine in France.

The brandy exported into this country as cognac similarly exceeds the total production of genuine cognac in France.

Other fruits by similar processes give alcoholic distillates—apple, pear, peach, prune, giving apple brandy, etc.—but during the last twenty-five years any distilled spirit suitably coloured and flavoured is sold in the name and place of the old eau de vie de vin or brandy wine.

We have the following meanings of the word as used in trade:

1. Brandy, a distillate from wines grown in France, including " distillation des vins altérés ou malade."
2. Brandy, distillates from wines grown in any country.
3. Brandy, distillates from fruits other than grape, which may or may not have the special characters indicative of their origin.
4. Brandy, distillates from grains, roots, molasses, etc. ("trade brandy ").

In 1850 the trade brandy amounted to one-tenth of the total wine and fruit brandy.

In 1893 the trade brandy amounted to ten times the amount of the wine and fruit brandy. These facts are well shown in the following table:

STATISTICS OF FRENCH BRANDY FROM 1840 TO 1900.

(The numbers in each of these tables refer to 1,000 gallons.)

	Natural (grape and other fruits).* (*a*)	Trade (maize, beet, etc.).† (*b*)	Total.	Approximate Proportion. (*a*) : (*b*)
1840	17,600	2,200	19,800	8 : 1
1850	17,950	1,661	19,611	10 : 1
1852‡	8,800	8,800	17,600	1 : 1
1860	—	—	10,206	—
1870	—	—	27,214	—
1876§	14,410	23,188	37,598	1 : 1·5
1880	1,100	34,100	35,100	1 : 30
1886	1,144	44,000	45,144	1 : 40
1893	5,434	49,060	54,494	1 : 10
1900	7,106	51,326	58,432	1 : 7

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* From French official sources, chiefly through the *Board of Trade Journal*. See also "Encyclopædia Britannica," vol. xxxii., p. 807.

† See also United States Consular Report, quoted in *Journ. Soc. Chem. Industry*, 1889, p. 827, referring to the very large exportation of "cognac" as compared with the total production of wine brandy.

‡ Oidium disease.

§ Phylloxera set in, 1876 to 1890.

In 1900 the following analysis is given of the production of brandy in France:

Wine, fruit, etc.	Wine	3,278
	Cider Lees, etc.	3,828
		7,106
Trade	Maize and other grains, etc.	12,386
	Molasses	17,534
	Beetroot	21,406
		51,326
	Total of all kinds	58,432

One-seventh of the total is exported as eau de vie de vin, but the amount so classed exceeds the total production from wine in the proportion of two to one. It also exceeds the total production from wine and other fruits in the proportion of seven to six. This is taking no account of the genuine eau de vie de vin used in France.

[NOTE.—It may be that part of this "trade brandy"—so termed in the official journals—is really alcohol destined for industrial purposes other than human consumption, and that these applications of spirit have much increased during the last thirty years. The returns I have been able to consult so far do not enable me to make allowance for these different applications of "trade brandy."]

The effect of this large sale of "trade brandy" as genuine eau de vie de vin is well shown in the following table of imports into the United Kingdom.*

1872	3,945
1882	3,013
1892	2,670
1902	2,355

The fall has been gradual and continuous, and the new certificates of origin issued by the French Government from March 30, 1903, are evidently a proof of a desire to regain trade by restoring confidence.

The following is a quotation from the *Distillers Brewers and Spirit Merchants Magazine*, referring to the proceedings of the Committee of the Wine and Spirit Association in a recent case: "If it were to go forth on the authority of the Association that 'brandy' is a meaningless term, applicable to any kind of spirit, the public would be less disposed than ever to ask for anything bearing that name."

To extend the meaning of the term "brandy" so as to include almost any alcoholic distillate is, in my opinion, calculated to absolutely destroy the confidence that a great part of the British public have had in the use of brandy, either as a superior spirit or as a medicinal agent. That this fear is well grounded is shown by the fact that, in spite of increased population and wealth, the importation and consumption of brandy has gone down about 40 per cent. during the last thirty years.

Some who defend the extension of the term "brandy" to other alcoholic distillates do not approve of the extension of the term "cognac," which they say should be strictly limited to the distillates from the wines of the Cognac district, and possibly of those districts immediately adjacent.

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* Abbreviated from the *Distillers Brewers and Spirit Merchants Magazine*, 1904, p. 232.

During the past twenty years a process of extension of the application of the term "cognac" has been going on, and it is now claimed that cognacs may be made in any part of France. This claim has been supported in a French court of law. Further, it is claimed that cognacs may be made in any other country where a product of a more or less similar character can be produced. We now have German and other cognacs.

In Murray's "New English Dictionary" now being issued it is stated that the term "cognac" is being "extended for trade purposes to mean brandy in general." This definition conforms with my own observation, both in this country and on the Continent.

I gladly thank Messrs. Scarisbrick, Nettleton, Squire, Stanley Smith, Murphy, and others for their kind assistance in collecting information; Mr. B. A. Burrell for kindly taking photographs and preparing slides, and Mr. Clifford for exhibiting the lantern slides.

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ON THE REDUCING ACTION OF HYDROGEN.

(Preliminary Note.)

By ALFRED C. CHAPMAN and H. D. LAW.

NOTWITHSTANDING the great theoretical and practical importance attaching to a more intimate knowledge of the properties of the hydrogen evolved during the solution of metals in acids, very little attention appears to have been devoted to this subject until quite recently, the employment of the expressions "nascent" and "free" being apparently considered sufficient to cover all the known facts. If the conceptions of *atomic* or *active* and *molecular* or *inactive* hydrogen were all-sufficient, the source of the hydrogen in any reduction process should be a matter of indifference so far as the reducing action is concerned; but yet it is quite well known that this is by no means the case, and that the reducing agent to be employed must be chosen in careful relation to the reduction which it is desired to bring about. This is well seen in connection with the Marsh-Berzelius test for arsenic, for not only does the hydrogen formed at the surface of a platinum cathode in the electrolytic method fail to reduce arsenates, but quite different results may be obtained in the zinc method by the employment of various samples of zinc. Before alluding to the results of our experiments, we may perhaps be permitted to refer very briefly to the following elementary theoretical considerations:

When a rod of zinc is immersed in dilute sulphuric acid, the zinc of course tends to pass into solution and to "force out" the hydrogen. On the other hand, a hydrogen electrode placed in a solution of zinc sulphate tends to cause the deposition of metallic zinc. Thus in the case we are considering (dilute sulphuric acid and zinc) hydrogen will be liberated only when the solution tension of the metal is greater than that of hydrogen. The solution tension of hydrogen is about 1 volt, whilst that of zinc is about 1·7 volts, so that an evolution of hydrogen might be expected. There is, however, another important factor which comes into play—viz., the "retaining" effect which every metal exerts on the hydrogen formed at its surface—and so in many cases a potential far in excess of that of the hydrogen electrode is necessary before any *free* hydrogen is obtained—that is, before the hydrogen is liberated. This excess of potential is known as "supertension," and in the case of zinc amounts to about 0·7 volt above that of the hydrogen electrode. From this it will be seen that in the case of pure zinc and sulphuric acid little or no evolution of hydrogen is to be expected. Other metals, however—*e.g.*, platinum—have smaller supertensions, and consequently small quantities of such metals deposited on the surface of the pure zinc will allow the gas to come off freely and at a lower potential, its activity being in consequence sometimes reduced to a point which renders it incapable of bringing about the reduction of difficultly reducible substances. The application of these considerations to the explanation of the insensitiveness of zinc in the Marsh-Berzelius test will, we think, be obvious. The metal, as used in our laboratories, is necessarily more or less impure, frequently containing particles of metals of low supertension—such, for example, as iron. Hydrogen is therefore liberated rapidly, and at a potential too low to permit of the reduction of arsenious acid to arsenic hydride under the conditions of the experiment. Thus we have been able to show that such low supertension metals as palladium, platinum, copper, nickel, cobalt, silver, and iron all render zinc insensitive, whilst cadmium, lead, and tin, which are possessed of high supertensions, have no effect on the reducing power of sensitive zinc. Starting with a sensitive sample of this metal, we have been able to render it exceedingly insensitive by depositing on its surface small quantities of iron, cobalt, etc.; and, conversely, we have succeeded in rendering the highly insensitive metal fully sensitive by the use of metals of high supertension. In the near future we hope to publish a detailed account of our work in this field. Throughout the investigation the Marsh-Berzelius arsenic process has been used merely as a convenient and delicate method of determining the amount of reduction taking place, and in a future paper we hope to extend the inquiry to other and widely differing substances.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

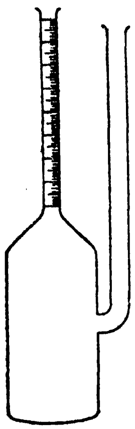
FOODS AND DRUGS ANALYSIS.

Detection of Formaldehyde in Milk. Utz. (*Chem. Zeit.*, 1905, xxix., 669.) —A reagent, consisting of vanillin and hydrochloric acid, is recommended for the detection of formaldehyde in milk. This reagent has been previously used by Winckel as a test for enzymes. Equal volumes of milk and hydrochloric acid (specific gravity 1·19) together with a few crystals of vanillin, are mixed and heated. Should formaldehyde be present in the milk, a violet or raspberry-red coloration appears. If the milk contain mere traces of formaldehyde, the mixture is coloured yellow. Similar reactions can be obtained by substituting piperonal or *p*-hydroxybenzaldehyde for the vanillin, but the latter is the best.

The author also shows that the presence of formaldehyde only interferes with reactions employed to distinguish raw from heated milk when the quantity of formaldehyde is largely in excess of that usually used for preservative purposes.

W. P. S.

Determination of Fat in Infant and Invalid Foods. C. B. Cochran. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 906-909.)—The determination is carried out in a flask of the construction shown in the figure. 1·72 grams of the powdered food are introduced through the side tube with 5 c.c. of water. After shaking, 2·5 c.c. of 80 per cent. acetic acid and an equal volume of concentrated sulphuric acid are added, and the contents of the flask well mixed. The flask is then placed in hot water until the contents turn a coffee colour. It is now removed and cooled, 4 c.c. of ether added, mixed, and boiled off by again placing the flask in warm water. The layer of fat floating on the surface of the liquid in the flask is brought into the graduated neck of the latter by pouring in hot water through the side tube. The volume of fat is then read off. The graduations give direct percentages when 5 c.c. of milk are used, so that in using 1·72 grams (one-third of the weight of 5 c.c. of milk) of the food the result must be multiplied by 3 to obtain the percentage of fat in the food. The apparatus and process may also be employed for the determination of fat in condensed milk, one part of the latter being mixed with four parts by weight of water before proceeding with the determination.



W. P. S.

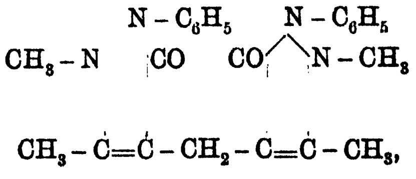
Corned Beef containing Flour. H. Matthes. (*Zeit. Untersuch. Nahr. Genussmittel*, 1905, ix., 732.)—During the examination of a number of tins of corned beef one sample was found to contain about 1·5 per cent. of flour. This was probably added to "bind" the pieces of beef together, and the author points out that, whilst fresh, tender beef readily forms a solid block of corned beef, tough beef from old animals requires the addition of some such material as flour. None of the samples were found to contain preservatives. W. P. S.

The Detection of Artificial Colour in Mustard. P. Süss. (*Pharm. Zentralh.*, 1905, xlvi., 291; *Chem. Zeit.*, 1905, xxix. [*Rep.*], 123.)—About 50 grams of the mustard are mixed with about 75 c.c. of 70 per cent. alcohol, allowed to stand for ten minutes, and filtered. A piece of unmordanted wool yarn is dipped in one portion of the filtrate, and then washed and dried in a porcelain basin over a small flame. Another portion of the filtrate is used for dyeing a "capillary strip" of the wool, which is immersed for twenty-four hours in the liquid and then dried. If the wool fibre shows a dirty yellow colour which speedily fades, artificial coloration is indicated, even when moistening the wool with hydrochloric acid or ammonia solution does not give a reaction—bluish-red (methyl orange), brownish-red (turmeric). A coal-tar dyestuff, which the author was unable to identify, dyed the wool lemon-yellow. In the case of the capillary strip, yellow edges are only observed when a coal-tar dyestuff is present. The usual tests with hydrochloric acid, ammonia, and boric acid must also be made here. The natural colouring matter of mustard either gives no coloration with hydrochloric acid, or at most a faint brown shade. It becomes intensely yellow on the addition of ammonia. C. A. M.

Occurrence of Boric Acid in Common Salt. R. Hefelmann. *Zeit. öffentl. Chem.*, 1905, xi., 231-234.)—Although some common salt, more particularly that mined in Italy and certain districts in Germany, undoubtedly contains boric acid, the quantity of the latter present is extremely small, varying from 0·0006 to 0·003 per cent. When this salt is used for flavouring meats or for salting butter and margarine, the amount of boric acid thus introduced is too small to be detected. W. P. S.

The Detection of Iodoform. Stortenbeker. (*Rec. Trav. Chim. des Pays-Bas*, 1905, xxiv., 67; *Chem. Zeit.*, 1905, xxix. [*Rep.*], 197.)—The following method is recommended for the detection of traces of iodoform in organs of the body: The substance is rendered slightly acid and distilled in a current of hydrogen. The distillate is treated with a few drops of alkali solution to dissolve any fatty acid present, which might interfere with the subsequent crystallization, and then shaken out with ether. The ethereal extract is allowed to evaporate spontaneously in the dark, and the residual drop of water taken up with filter-paper. The residue is now dissolved in a little hot glacial acetic acid, and the residue left on evaporation of a portion of the solution examined under the microscope. Well-marked crystals are obtained in the presence of iodoform. The colour reaction given by iodoform with sodium phenolate is not interfered with by the presence of chloroform or bromoform. C. A. M.

A New Method of Analyzing Pyramidon. G. Pattein. (*Bull. Soc. Chim.*, 1905, xxiii., 845-847.)—This is based upon the fact that pyramidon (dimethylamido-antipyrin) gives no insoluble derivative with formaldehyde, whereas antipyrin gives a nearly insoluble compound—



which the author terms diantipyrin methane. If 1 gram of pure pyramidon be treated with 5 c.c. of water, 5 c.c. of hydrochloric acid, and 2 c.c. of a 40 per cent. solution of formaldehyde, and left in a corked tube for four days, the liquid remains absolutely clear after the addition of 10 c.c. of water and sufficient ammonia to make the reaction alkaline. But if antipyrin is present a crystalline deposit (melting point 177° to 179° C.) is formed as the liquid cools. It can be collected on a filter, dried in the oven, and weighed. Theoretically, 0·2 gram of antipyrin should yield 0·214 gram of the compound, but in practice the amount varies from 0·18 to 0·20 gram. In the case of a mixture the filtrate from the crystals can be extracted with chloroform, and the extract allowed to evaporate spontaneously, when crystals of uncombined pyramidon (melting point 104° to 106° C.) will be left. If time is limited the tube may be left for four hours on the water-bath at 100° C., and the liquid then cooled and treated as above described. C. A. M.

The "Manipulated" Opiums of Smyrna. V. Masson. (*Journ. Pharm. Chim.*, 1905, xxi., 529-534.)—For several years past certain dealers have supplied opiums manufactured at Smyrna in place of the natural product. These opiums are stated by the author to be extensively met with in commerce under the name of Smyrna opium, 10 per cent., or "manipulated" opium, and consist of a mixture of the natural juice with substances more or less inert. The proportion of foreign substances varies considerably, and alters considerably the composition of the components that contain morphine as their base. The products differ in physical characteristics from natural opiums, and may be described as pastes, the only standard of which is that they contain not less than 10 per cent. of morphine. They give an alkaline reaction in contradistinction to true opiums which give an acid one, and the whole of the morphine they contain does not pass into the aqueous extract. This may be owing to a portion of the morphine meconate having been decomposed by added calcium carbonate, or to morphine, and not one of its salts, having been added to an exhausted marc. The following analyses represent the compositions of five different types of these preparations and of samples of pure opium examined by the author. The extract is calculated on the basis of opium containing 10 per cent. of water, whilst the morphine is calculated on the substance dried at 100° C.

	Water. Per Cent.	Aqueous Extract. Per Cent.	Morphine. Per Cent.	Ash. Per Cent.	Morphine in Aqueous Extract. Per Cent.
Smyrna manipulated opium, 1	14·5	20·0	10·2	—	—
,, ,, ,, 2	9·24	20·20	13·0	5·88	22·28
,, ,, ,, 3	8·31	27·80	12·19	5·71	29·96
,, ,, ,, 4	19·11	44·52	10·62	9·25	18·67
,, ,, ,, 5	21·17	57·30	10·12	5·05	12·0
Pure opium, Yerli	26·1	53·95	13·51	—	
,, Salonica	24·97	49·17	12·18	—	
	17·62	46·35	10·53		
Smyrna	20·93	44·83	10·82		

The gum in the extract of No. 5 was 21·50 per cent., as against 2·50 per cent. in natural opium. C. A. M.

A Method of Distinguishing Dionin from Codein. N. D. Rudionon. (*Chem. Zeit. Rep.*, 1905, xxix., 187.)—Dionin (hydrochloride of mono-ethylmorphine) has the same colour reactions as codeine (mono-methylmorphine), but their dissimilar behaviour towards ammonia may be used to distinguish between the two. The author also finds a difference in their reactions with Wagner's reagent (12·7 grams of iodine and 10 grams potassium iodide in 1,000 c.c. of water). When 10 drops of this reagent are added to 2 c.c. of a 1 per cent. hydrochloric acid solution of codeine, a pulverulent dark red-brown precipitate is obtained. On violent shaking this precipitate does not change its colour, and immediately settles again to the bottom. If a 1 per cent. dionin solution is treated in the same way, a precipitate of the same kind and colour is obtained; but on vigorous shaking it becomes flocculent, acquires a brown-orange colour, and rises to the surface of the liquid. The reactions take place in neutral as well as in sulphuric or hydrochloric acid solutions. As very vigorous shaking is necessary, it is best to use test-tubes with glass stoppers.

E. K. H.

Identification of Rheum Rhaponticum. A. Tschirch. (*Schweiz. Wochenschr.*, xliii., 253.)—The rhizome of *R. rhaponticum* (common English rhubarb) contains a crystalline substance, called rhaponticin, rhapontin, or ponticin, which is characterized by its insolubility in ether, separating from its aqueous solution when shaken with that liquid. The following method for detecting *R. rhaponticum* is based on this peculiarity: Ten grams of the sample are boiled with 50 c.c. of dilute alcohol for fifteen minutes, then filtered, and the filtrate concentrated to about 10 c.c. The latter is shaken with from 10 to 15 c.c. of ether. On allowing the mixture to stand, a considerable quantity of a minute crystalline deposit will be formed if *R. rhaponticum* be present. The deposit, after being collected on a filter, washed with water, and dried, is coloured purple-red by sulphuric acid. Chinese rhubarb, similarly treated, gives no such crystalline deposit.

W. P. S.

ORGANIC ANALYSIS.

Detection of Methyl Alcohol. H. Scudder. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 892-906.)—In comparing the tests for methyl alcohol, the author takes as the standard of delicacy that quantity of methyl alcohol which can be detected in ethyl alcohol. Most of the tests will show very much smaller quantities of methyl alcohol in water, but their use for such a purpose is practically unknown. The Mulliken-Scudder test (ANALYST, 1901, 74) will show the presence of 8 to 10 per cent. of methyl alcohol, whilst the United States Pharmacopœia test will indicate 2 per cent. Trillat's test (ANALYST, 1899, 211) is somewhat more delicate. If only traces of methyl alcohol be present, it is advisable to concentrate by fractional distillation before testing. The value of the resorcinol (Mulliken-Scudder) test is in the positive evidence given, and the advantage of having a rough idea of the quantity of formaldehyde present. In testing mixtures, any formaldehyde present must be previously removed. For this purpose various methods may be employed (see ANALYST, 1891, 113; 1901, 74). Other aldehydes are removed by digestion with resorcinol and sulphuric acid, or aniline and phosphoric acid. Phenols and bases will combine with any formaldehyde produced in the course of the test, and must be removed by distillation with potassium hydroxide in the case of phenols, and with sulphuric acid in the case of bases. Colouring matters can be removed by distillation or filtration through animal charcoal.

W. P. S.

Determination of Glycerol in Crude Glycerines, etc. K. Braun. (*Chem. Zeit.*, 1905, xxix., 763-765.)—In the process described the glycerol is oxidized by means of potassium bichromate (chromic acid), and the resulting carbon dioxide collected and determined. A weighed portion of the sample is diluted with water. Should carbonates be present, they must be decomposed by boiling the diluted and acidified solution. The latter is then made up to a known volume. A measured quantity of this solution is transferred to a round-bottomed flask of about 1 litre capacity, an excess of standard bichromate solution is added, and also about 10 c.c. of sulphuric acid. The flask is connected with a Liebig's condenser, the lower end of which is drawn out and passed into a receiver containing a known volume of $\frac{N}{1}$ potassium hydroxide solution. A small flame is used for heating the flask, and the distillation carried on for about thirty minutes. Hot barium chloride solution is then added to the contents of the receiver, the precipitated barium carbonate being collected on a filter and washed until free from alkali. Both filter and precipitate are now placed in a flask, more than sufficient $\frac{N}{10}$ hydrochloric acid to dissolve the barium carbonate is added, and the excess titrated back with $\frac{N}{10}$ potassium hydroxide. One c.c. of $\frac{N}{10}$ acid is equivalent to 0·001533 gram of glycerol. The method is stated to give results which agree well with those obtained by other methods.

W. P. S.

Gasometric Determination of Formaldehyde. G. B. Frankforter and R. West. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 714-719.)—The process described is based on the fact that when formaldehyde is brought into contact with sodium or potassium hydroxides in the presence of hydrogen peroxide, potassium formate is formed and hydrogen liberated according to the formula—

$$2CH_2O + 2KOH = 2HCOOK + 2H_2O + H_2.$$

In the absence of hydrogen peroxide, methyl alcohol is formed and no hydrogen set free. The authors measure the volume of the liberated hydrogen, and from it calculate the amount of formaldehyde present. The details of the process are: 1 c.c. of the formaldehyde solution of known specific gravity is placed in the side tube of a Schiebler-Finkner calcimeter, and 10 c.c. of hydrogen peroxide and 20 c.c. of $\frac{N}{1}$ potassium hydroxide solution are placed in the body of the generator. The latter is closed, and the water in the measuring-tube brought to the zero mark. After the temperature has become constant, the solutions are gradually mixed. When the reaction is completed the temperature is again allowed to become constant, and the volume of hydrogen read off. The presence of alcohol and formic acid has no influence on the results, provided that a good excess of hydrogen peroxide be present. Sodium peroxide previously dissolved in water may be used instead of hydrogen peroxide, but slightly too high results are obtained, probably due to dissolved oxygen. With barium peroxide the reaction is slow and incomplete in the cold. Manganese and lead peroxides are practically without action on formaldehyde. The authors describe experiments to prove that the gas liberated in the above reaction consists of pure hydrogen.

W. P. S.

The Polarimetric Determination of Sucrose. F. Watts and H. A. Tempany. (*W. Indian Bull.*, 1905, vi., 52-60.)—The authors have investigated the influence of alterations of temperature on the optical activity of sucrose. They find that changes of temperature, up to about 30° C., cause a lowering of the reading on the Ventzke scale which may be corrected by the formula : Polarization $+(0{\cdot}00023t)$N, where t is the difference of temperature of observation from that at which the instrument was standardized, and N is the scale reading observed. A correction must also be made for the influence of temperature on the quartz wedge of Schmidt and Haensch instruments, and may be expressed by the formula (Jobin's): Polarization $+(0{\cdot}00016t)$N; or the two corrections may be combined: Polarization $+(0{\cdot}00039t)$N. Of course, where the temperature of observation is below that at which the instrument was standardized, the correction becomes a negative one.

For solutions requiring no clarifying reagents the authors recommend that 26 grams of the sugar be dissolved in 100 *true* c.c. This is equivalent to 26·048 grams in 100 Mohr c.c. The true cubic centimetre is taken as being the volume of 1 gram of water weighed *in vacuo* at 4° C., whilst 100 c.c. Mohr are defined as being 100 grams of water at 17·5° C., in air, no correction being made for displacement of air ; under these conditions 100 c.c. Mohr become equal to 100·228 c.c. true.

The correction of the volume of lead precipitate in solutions which require clarifying varies for almost each class of sugar, owing to the varying density of the precipitate. It is better, instead of adding basic lead acetate solution to the sugar solution and diluting the mixture to a known volume, to dissolve 26 grams of the sugar in distilled water, make up to 100 true c.c., and clarify by adding anhydrous basic lead acetate, avoiding an excess. W. P. S.

Note on a Fluid Portion obtained from Cacao Butter. F. Strube. (*Zeit. öffentl. Chem.*, 1905, xi., 215, 216.)—By slowly cooling large blocks of "Samana" cacao butter, a fluid portion occasionally separated out, which on examination yielded chemical and physical constants differing considerably from those given by ordinary cacao butter. For instance, various specimens of this fluid portion were found to have iodine values of from 53·08 to 58·80, and gave refractometer readings of 50·45 at 40° C. The melting-point was very low—about 12° C.; at 17·5° C. the specific gravity of the specimens was 0·906. W. P. S.

The Detection of Oil extracted by Carbon Bisulphide in Expressed Olive Oil. G. Halphen. (*Journ. Pharm. Chim.*, 1905, xxi., 54-56.)—The method depends on the conversion of residual sulphur compounds into thiosulphates, which remain in solution when the soap is salted out by sodium sulphate or chloride. Fifty c.c. of the olive oil are heated to 110° C. in a basin, and treated with 12 c.c. of a solution of 100 grams of pure sodium hydroxide in 75 c.c. of water. The heating is then continued for about seven to ten minutes until the mass ceases to froth, the temperature being then about 160° C. The flame is now withdrawn and the mass continually stirred until the temperature has fallen to 110° C. and the soap is finely granulated. It is then treated with 200 c.c. of hot water, and constantly shaken until cold, when practically the whole of the soap should have dissolved. This solution is shaken with 100 c.c. of a saturated solution of sodium sulphate, after which 20 c.c. of a 33 per cent. solution of copper sulphate are introduced, and the liquid thoroughly stirred and filtered. The object of adding the copper sulphate is to convert into insoluble copper salts impurities in certain expressed olive oils, which give a slight brown precipitate with silver nitrate. The filtrate should be faintly green, and, if not, it should be treated with an additional 0·2 c.c. of the copper sulphate solution and again filtered. To 100 c.c. of the clear filtrate are added 5 c.c. of a solution consisting of 1 part of a 5 per cent. solution of silver nitrate with 5 parts by volume of glacial acetic acid, and the whole gently heated to the boiling-point, then cooled, and rendered slightly alkaline with ammonia. If the oil had been extracted with carbon bisulphide a brown or black precipitate will be formed, although in some cases it is necessary to collect it on a filter before it can be detected. The author states that the black precipitate is invariably obtained even when only small quantities of the extracted oil are present. It is, of course, necessary to insure the absence of the oils of cruciferous plants, which normally contain sulphur. C. A. M.

The Refractometer Values of Castor Oil, Cod-liver Oil, and Neat's Foot Oil. H. C. Lythgoe. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 887-892.)—The following results were obtained from the examination of a number of pure samples of these oils :

	Castor Oil.	Cod-liver Oil.	Neat's Foot Oil.
Number of samples	44	20	4
Average butyro-refractometer (Zeiss) reading at 20° C.	81·2	80·6	63·5
Maximum variation of reading ...	±0·5	±1·3	±0·2

The average polarization of fifteen samples of castor oil, when read in a 200-millimetre tube at 20° C., was +24·1° Ventzke. Tables are also given showing the refractometer values of these oils for each 0·5 degree of temperature from 15·0° to 35·0° C. W. P. S.

A New Reaction of Substances containing a Phenolic Group. J. Aloy and F. Laprade. (*Bull. Soc. Chim.*, 1905, xxxiii., 860, 861.)—Uranyl nitrate gives a coloration, usually red, with bodies containing a phenolic group. The reagent is prepared by dissolving 10 grams of uranyl nitrate in about 60 c.c. of water, adding a dilute solution of ammonia until the liquid is exactly neutral, filtering it, and diluting the filtrate to 100 c.c. Other uranyl salts can be used in place of the nitrate, but it is essential to neutralize the solution exactly. In testing for a phenoloid substance the liquid is first neutralized and the reagent added drop by drop until the intensity of colour no longer increases. The presence of one COOH, NH_2 or NO_2 group does not interfere with the reaction, but several groups in the molecule may prevent it. The colour is destroyed by mineral acids and by alkalies. The reaction is capable of detecting 1 part of phenol or less in 1,000. C. A. M.

On the Determination of Nitrogen by Kjeldahl's Method. S. P. L. Sorensen and C. Pedersen. (*Comptes Rend. des Trav. du Lab. de Carlsberg*, 1905, vi., 126-136.)—It has been asserted by Kutscher and Steudel that Kjeldahl's method is not applicable in the case of certain compounds, such as creatine, creatinine, lysine, and uric acid. The authors have therefore made experiments to determine this point. They show that one source of error on the part of Kjeldahl's critics is that, in the oxidation with potassium permanganate, they add this salt to the cooled liquid, which is then heated to the boiling-point. This is quite contrary to Kjeldahl's directions, and makes a loss of ammonia inevitable. The results show that the nitrogen in creatine is converted rapidly and completely into ammonium salts when boiled with concentrated sulphuric acid, but that lysine compounds are only transformed slowly even after the addition of phosphotungstic acid, as advocated by Henderson. The general conclusion is that Kjeldahl's method is quite applicable to these compounds, although the results are very slightly too low. Thus, the "racemic chloride of lysine" gave 12·63 per cent. of nitrogen by Dumas' method, and 12·52 per cent. by Kjeldahl's method. (*c.f.* Abstract, 1904, xxix., 124.) C. A. M.

On the Determination of Nitrogen in Lysine and Similar Compounds. S. P. L. Sorensen and A. C. Andersen. (*Comptes Rend. des Trav. du Lab. de Carlsberg*, 1905, vi., 193-208.)—Certain derivatives of lysine are particularly resistant to the action of sulphuric acid. Thus, lysuric acid, after having been boiled for twenty-four hours, yielded only 7·56 per cent. of nitrogen by Kjeldahl's method, as against the theoretical 7·93 per cent. Hence it is recommended that in the analysis of the decomposition products of proteids parallel determinations should be made by Kjeldahl's and by the Gunning-Arnold method; and should concordant results be obtained, only the former simpler method need be used subsequently. If, however, too little ammonia is obtained, there must be present compounds only decomposed with difficulty, and in which the nitrogen is in ring-formed combination—*e.g.*, derivatives of pyridine or piperidine. The compounds may thus be divided into two groups —viz., (*A*) compounds containing or capable of forming five membered rings, composed of 4 atoms of carbon and 1 of nitrogen. All these can be analysed by Kjeldahl's method—*e.g.*, α-pyrrolidine carboxylic acid, α-amino-δ-hydroxyvaleric acid, α-δ-diamino-adipic acid; (*B*) compounds containing or capable of forming six membered rings, consisting of 5 atoms of carbon and 1 of nitrogen—*e.g.*, pyridine, piperidine, lysuric acid, α-ϵ-diaminopimelic acid. The last-named compound forms a piperidine ring when boiled with strong sulphuric acid, and behaves like piperidine itself, yielding lower results by Kjeldahl's method than by the modifications of Gunning, or Gunning and Arnold. C. A. M.

The Determination of Nitrogen in Azo- and Similar Compounds by Kjeldahl's Method. C. Flamand and B. Prager. (*Berichte*, 1905, xxxviii., 559, 560.)—From 0·15 to 0·2 gram of the substance is dissolved in 10 c.c. of alcohol in a 500 c.c. flask with a long neck, and the solution heated with 0·5 to 1 gram. of zinc dust and 2 to 5 c.c. of sulphuric acid (specific gravity 1·19) until colourless, which usually takes a few minutes. Ten c.c. more of the concentrated sulphuric acid and 0·5 gram of crystallized copper sulphate are then introduced, and the flask heated until white fumes appear, after which 5 grams of potassium sulphate are added and the heating continued until the liquid has become clear and light green in colour, which is usually the case after fifteen minutes. The ammonia is then distilled in the usual way. Phenyl-hydrazine, benzalphenyl-hydrazine, and formazyl compounds did not give satisfactory results by this method, although the theoretical amount of nitrogen was obtained in the case of various azo-, hydrazo-, and azoxy-derivatives.

C. A. M.

Determination of the Organic Nitrogen in Potable Waters. J. C. Brown. (*Proc. Chem. Soc.*, 1905, xxi., 208, 209.)—Details are given of a process for the determination of the whole of the organic nitrogen in water and sewage effluents. The process essentially consists in the distillation to dryness and subsequent ignition of a mixture of a portion, usually 200 c.c., of the sample, without previous evaporation, with potassium hydroxide and potassium permanganate. The operation is carried out either in a Jena glass or in a copper retort, and the evolved ammonia is determined by Nessler's reagent as usual. The process is found to be convenient and rapid. The results obtained by this process on a variety of waters compare well with those determined by the Frankland, Wanklyn, and Forschammer methods. The total organic carbon may be determined as carbon dioxide in the residue remaining in the retort, but owing to the large amount of carbon dioxide found by blank experiments, and which has to be deducted from the total quantity, the results are not as accurate as in the case of nitrogen.

W. P. S.

The Determination of Alkaloids by Means of Potassium Bismuth Iodide. H. Thoms. (*Ber. deutsch. Pharm. Ges.*, 1905, 85; *Journ. Pharm. Chim.*, 1905, xxi., 605.)—The reagent is prepared by dissolving 80 grams of bismuth subnitrate in 200 grams of dilute nitric acid (specific gravity 1·18) and pouring the solution into an aqueous solution containing 272 grams of potassium iodide. The bulk of the potassium nitrate formed crystallizes out, and the decanted liquid is evaporated down to a litre.

For the valuation of extract of belladonna 2 grams of the sample are dissolved in 50 c.c. of water containing 10 c.c. of dilute sulphuric acid (1:10), and 5 c.c. of the reagent added little by little with continual stirring. The resulting precipitate is collected on a filter and washed twice with 5 c.c. of sulphuric acid (1:10). The filter and precipitate are then shaken in a stoppered flask with 0·3 gram of sodium sulphite and 30 c.c. of a 15 per cent. solution of sodium hydroxide until the alkaloids are liberated, after which 15 grams of sodium chloride and 100 c.c. of ether are introduced, and the shaking continued until the alkaloids have been taken up by the ether. The mixture is left to separate, 50 c.c. of the ethereal layer then removed by means of a pipette, and the amount of alkaloids it contains determined by titration with a $\frac{N}{100}$ hydrochloric acid, iodeosin being used as indicator. The number of c.c. used multiplied by 0·289 gives the weight of total alkaloids in terms of atropine. Test experiments showed that 97 per cent. of the alkaloid present was determined by this method.

C. A. M.

The Extraction of Tannin Materials with Various Extractors. F. P. Veitch. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 724-729.)—To prevent possible oxidation of tannin by prolonged boiling, and to provide for the extraction of certain materials from which maximum results are only obtained at low temperatures, a number of forms of apparatus have been used for the laboratory extraction of tanning materials. The two extractors at present in general use are the Koch and a modified Soxhlet, known as the Weiss extractor. With the Koch extractor, the extract, amounting to 2 to 4 litres, has to be evaporated, thus encouraging the very oxidation it was designed to prevent. The apparatus, however, enables the temperature during the extraction to be controlled. The Weiss extractor is used in most tannery laboratories in America for extracting woods and barks, but the prolonged boiling of the materials is regarded by some authorities as inadmissible. The author prefers to use a continuous extractor, and describes one shaped like an ordinary Soxhlet, but without the siphon tube and with the lower central tube in direct communication with the body of the apparatus. The powdered material is made into a paste with water and poured into the extractor, a perforated porcelain disc being placed at the bottom of the body of the extractor, and a similar disc on the top of the material. The turbid liquid which runs through at first is returned to the extractor, which is then connected with a condenser. The lower end of the condenser should reach below the side tube of the apparatus. The results obtained by these three extractors are compared, and show that the highest tannin results are yielded with the continuous extractor, except in the case of sumach, where the Koch apparatus gives the highest result. With the exception of sumach, there is nothing to indicate that there is any serious loss of tannin with any of the extractors. W. P. S.

The Determination of Ash in Vegetable Substances. E. Gutzeit. (*Chem. Zeit.*, 1905, xxix., 556.)—The powdered substance is intimately mixed with a known weight of freshly-ignited basic calcium phosphate and very gently heated in an open platinum crucible until the organic substance is carbonized, after which it is ignited at a moderate heat, eventually with the crucible partially covered, until the residue appears white, when it is weighed as rapidly as possible. The ash thus obtained is quite free from carbon particles and carbon dioxide. After each determination the crucible and its contents are ignited more strongly, and can then be used again for a new determination. The basic calcium phosphate is prepared by mixing pure finely-powdered calcium chloride and sodium phosphate in such quantities that the ratio of calcium oxide to phosphoric anhydride corresponds to the formula of basic calcium phosphate $3CaO.2P_2O_5[x OH ?]$, and igniting the mixture. The mass is digested with hot water, thoroughly washed, and dried. C. A. M.

INORGANIC ANALYSIS.

The Separation of Platinum and Iridium. L. Quennessen. (*Chem. News*, 1905, xcii., 29, 30.)—The alloy under examination is dissolved in aqua regia, composed of 1 part by volume of nitric acid of specific gravity 1·32, with 2 parts of hydrochloric acid of specific gravity 1·18, and the excess of nitric acid afterwards expelled by heating the solution, at first gently, and finally at 120° C. in an oven. The residue is then treated with water, the metals precipitated by means of magnesium, and the precipitate dried, ignited, and heated at a dull red heat in a current of hydrogen. The excess of magnesium is subsequently removed by means of dilute sulphuric acid (1 : 10), and the residue, treated with the aqua regia diluted with three times its volume of water. This dissolves only the platinum, which is then converted into the double chloride of ammonium, and ignited at as low a temperature as possible, so as to prevent appreciable loss in the form of platinous chloride. The use of a reducing agent, such as oxalic acid or glucose, is advisable, or the filter-paper containing the dried precipitate may be placed upside down in the covered crucible in the furnace, and the charred paper then acts as a reducing agent, its combustion being subsequently completed in the air after the operation is over. The iridium precipitated by the magnesium is soluble, while still in a moist condition, in acetic and dilute sulphuric acids, and does not lose this property when dried at 100° C. It imparts a yellow colour to the sulphuric acid, which gradually changes to violet, while the acetic acid solution is green. After being heated in the air to 440° C., the oxide gives a violet colour with sulphuric acid, and a blue colour when the temperature has been raised to 800° C. C. A. M.

On the Separation of Tungsten Trioxide and Silicon Dioxide by Means of Gaseous Hydrogen Chloride, and the Analysis of Silico-Tungstates. Carl Friedheim, W. H. Henderson, and Alfred Pinagel. (*Zeits. anorg. Chem.*, 1905, xlv., 396.)—Tungsten trioxide and silica may be separated by heating the dehydrated mixture contained in a platinum boat in a glass tube in a current of dry hydrogen chloride. The tungsten is volatilized, partly as chloride, which is caught in a receiver containing strong hydrochloric acid, and partly as tungstic acid, which sublimes into the cooler parts of the glass tube, and may be detached with a platinum wire. The silicon remains behind as dioxide. A fairly bright red heat is necessary for the separation, which proceeds the more slowly the more strongly the oxides have been heated previously. The method gives exceedingly exact results, as the test analyses quoted show, and is of especial use in those cases where the silica cannot be removed from the mixture by treatment with hydrofluoric acid—that is, when the anhydrides have been obtained by ignition of the mixed salts, *e.g.*, mercury or quinoline salts, of the two acids.

In applying the method to the analysis of silico-tungstates, the salt is evaporated to dryness with hydrochloric acid in a platinum boat, the residue being ignited and treated as above. The presence of potassium or sodium chloride does not interfere with the volatilization of the tungsten, but the platinum boat is seriously attacked, especially with potassium salts, if the hydrogen chloride carries any air with it. On the other hand, if no air is present, the tungsten trioxide occasionally suffers reduction, and is then not further acted on by the hydrogen chloride. It is, therefore, best to fit the combustion tube with a T-inlet tube, through which either dry hydrogen chloride or air may be introduced. The reaction is carried out as far as possible in air-free hydrogen chloride. Should the mixture in the boat become green, indicating reduction of the tungsten trioxide, the current of gas is stopped, the heating interrupted, and air led through the tube for some little time, after which the air is shut off, and the heating in hydrogen chloride resumed. The residual silica in the boat is weighed after evaporating to dryness with acid and extraction of the residue with acid. As it may contain platinum, it is then volatilized with hydrofluoric acid. The tungsten is determined as usual in the liquid in the receiver. A. G. L.

Rapid Method for the Estimation of Potash. E. H. Schultze. (*Chem. Zeit.*, 1905, xxix., 509.)—The method is one especially adapted for kainite and the so-called "40 per cent. salt." It is carried out as follows: Twenty grams of kainite or 10 grams of "40 per cent. salt" are boiled in a 500 c.c. flask with about 250 c.c. water and 10 c.c. hydrochloric acid for ten minutes. The sulphuric acid is then precipitated by a not too large excess of barium chloride whilst boiling, and the boiling continued for one minute. For kainite 100 c.c., and for "40 per cent. salt" 10 c.c., of 10 per cent. barium chloride should be used. The precipitate is allowed to settle, and then a few drops of barium chloride are added. If no further precipitate is caused, the process is continued; if a precipitate again forms, 20 c.c. more barium solution are added and the liquid again boiled. When the whole of the sulphate has been precipitated, the solution is cooled, made up to 500 c.c., shaken, and then filtered; 10 c.c. of the filtrate (=0·4 gram kainite or 0·2 gram "40 per cent. salt") are treated in a beaker of 300 to 400 c.c. capacity with 5 c.c. of a solution of platinic chloride (10 c.c. = 1 gram platinum), and 120 c.c. of hot 96 per cent. alcohol added in small portions. It is then stirred continuously for fifteen minutes, the beaker being placed in an outer vessel through which a current of water flows. This method, in addition to securing purity and completeness of the precipitation, results in the formation of the largest possible crystals. Immediately after the stirring (more easily after the precipitate has settled) it can be filtered off. It is washed with 82 per cent. of alcohol, containing a little ether, and then dried to constant weight in an oven at about 120° C.

E. K. H.

On the Determination of Ammonia in Water. Cavalier and Artus. (*Bull. Soc. Chim.*, 1905, xxxiii., 745-747.)—Quantitative experiments with the nitrogen iodide method of Trillat and Turchet (ANALYST, xxx., 218, 273) have shown that the new method is much less sensitive than that of Nessler, no coloration being perceptible until the proportion of ammonia is as much as 3 mgms. per litre. Hence, in the case of drinking-water containing only 0·2 mgm., it is necessary to evaporate 500 c.c. of the water (in the presence of a small amount of sulphuric acid) in order to obtain the 20 c.c. required for a colorimetric determination; and this evaporation is as tedious as the distillation in Nessler's process. A more serious drawback is that the black coloration of the iodide is very unstable. After the lapse of a minute the colour of water containing 10 mgms. is fainter than that immediately given by water containing 5 mgms., and it disappears almost completely after two or three minutes.

C. A. M.

The Reduction of Chlorates, Bromates, and Iodates for the Determination of the Halogen. P. Jannasch and A. Jahn. (*Berichte*, 1905, xxxviii., 1576-1592.)—Chlorates and bromates are reduced quantitatively by fuming nitric acid, but iodates are only reduced when heated with it under pressure. Hydrogen peroxide cannot be used as a reducing agent in an alkaline solution, but if used in a dilute nitric acid solution it gives a quantitative reduction of chlorates, certain precautions being necessary. Bromates are also reduced (though the nitric acid possibly plays a part in the process), but there is no reduction in the case of iodates. Iodates and bromates are completely reduced by hydrazine sulphate in alkaline solution, but not chlorates, and formic acid has a similar action. Neither acetaldehyde nor dextrose effect reduction either in a nitric acid or acetic acid solution, but chlorates are quantitatively reduced when heated under pressure with dextrose and acetic acid. The best results are obtained by the use of hydroxylamine sulphate, which effects the reduction in either an acid or ammoniacal solution, the latter being preferable for iodates. In determining the halogen in chlorates or bromates about 0·25 gram of the salt is dissolved in 50 c.c. of water and treated with 5 grams of hydroxylamine sulphate, after which the liquid is rendered strongly acid with nitric acid and heated, and the halogen precipitated by means of silver nitrate. Other salts of hydroxylamine or hydrazine, such as the acetate, can be used so as to eliminate the disturbing influence of the sulphates on the precipitation of silver chloride. When the reduction is to be effected in an alkaline solution an excess of ammonia solution is introduced prior to the addition of the hydroxylamine salt.

C. A. M.

Volumetric Determination of Nitrous Acid by Means of Ceric Sulphate. G. Barbieri. (*Chem. Zeit.*, 1905, xxix., 668, 669.)—Nitrous acid is completely oxidized in the cold by ceric salts, according to the equation—

$$2Ce(SO_4)_2 + KNO_2 + H_2O = Ce_2(SO_4)_3 + KNO_3 + H_2SO_4.$$

The most accurate results are obtained when the nitrite solution is treated with more than the required quantity of ceric sulphate solution, the excess of the latter being afterwards decomposed by the addition of potassium iodide and the liberated iodine titrated as usual. The ceric sulphate solution must be previously titrated to ascertain its oxidizing value. The presence of considerable quantities of nitrates does not interfere with the reaction, nor is it necessary that the ceric sulphate be free from lanthanum, praseodymium or neodymium. W. P. S.

Rapid Method for the Estimation of Phosphoric Acid. E. H. Schultze. (*Chem. Zeit.*, 1905, xxix., 509.)—The process depends upon the solubility of ammonium phosphomolybdate in alkalies. Solutions of potassium hydroxide are used, approximately normal in strength, and these are standardized against phosphate solutions of known strength. The analysis is carried out as follows: A solution of the substance is obtained in the case of superphosphate by simply shaking 10 grams of the substance with 1,000 c.c. of water. Insoluble phosphates are dissolved in dilute sulphuric acid in the usual way. A quantity of the solution, corresponding to from 0·2 gram to 1·2 grams of the substance, according to its expected percentage of phosphoric acid, is run into an Erlenmeyer flask, and potash solution added till a turbidity is just produced. Fifteen c.c. of nitric acid (specific gravity 1·40) and

20 c.c. ammonium nitrate (1 : 2) are added, and the whole made up to 80 to 100 c.c., and heated over wire gauze to about 80° C. To the hot solution 40 c.c. of the molybdate solution *A* are added, the mixture shaken up, and kept on a water-bath at 80° C. for five minutes. After filtration, the precipitate is washed by decantation once with water containing 1 per cent. nitric acid, and then twice with cold water, it is then transferred to the filter without removing the last residues in the Erlenmeyer, and further washed six to eight times. The filter funnel is then placed on the Erlenmeyer flask in which the precipitation was first made, the filter-paper pierced, and the precipitate washed into the flask, and the filter finally put into the flask also. Exactly 25 c.c. of the normal KOH solution, which has been standardized against phosphoric acid solution, are then run in from a burette, the flask is shaken up to get the whole of the precipitate into solution, and then the excess of KOH estimated by normal HCl, using phenolphthalein as indicator. Knowing the phosphoric acid equivalent of the potash solution (for the dissolving of ammonium phosphomolybdate) by the standardization experiment, the percentage of phosphoric acid in the sample can be calculated. Solution *A* is made by dissolving 800 to 900 grams of ammonium molybdate in 1,700 to 1,800 grams concentrated ammonia (specific gravity 0·91) and 10 litres of water, and then pouring into 10 litres concentrated nitric acid. It is filtered after standing for one day. E. K. H.

On the Alleged Volatility of Silicon Dioxide at the Instant of Precipitation by a Strong Acid. Carl Friedheim and Alfred Pinagel. (*Zeits. anorg. Chem.*, 1905, xlv., 410.)—Kehrmann and Flürscheim (*Zeits. anorg. Chem.*, xxxix., 98 and 106) have ascribed some losses in silica found in their analyses of silico-tungstates to a possible volatility of silicon dioxide at the instant when it is precipitated from its salts by hydrochloric or nitric acid. To test this assumption, they fused some ignited silica with potassium sodium carbonate, and evaporated the aqueous solution of the melt twenty times with concentrated hydrochloric acid. In this way a loss of 1·59 per cent. of the silica was found. The present authors have made three similar experiments to check Kehrmann and Flürscheim's statement. From the results obtained they conclude that it is entirely without foundation, and that the losses of silica in question were probably caused by dehydrating the silica at too low a temperature (at 100° C.), and by washing it finally with pure water instead of using dilute acid. A. G. L.

APPARATUS.

An Electrical Method for the Combustion of Organic Compounds. H. N. Morse and L. S. Taylor. (*Amer. Chem. Journ.*, 1905, xxxiii., 591.)—The apparatus described is strongly recommended as being very compact, wasting but little heat, and allowing a combustion to be made in much less time than in the ordinary way. In Fig. 1 the simplest form of the apparatus is represented. A glass combustion-tube, closed at one end, of a length of 350 mms. and an internal diameter of 15 mms., is fitted with a rubber stopper at its open end. Through this stopper there pass: the porcelain tube *c*, 250 mms. long and 6 mms. in diameter; the glass tube *k*, through which the products of the combustion enter the absorption apparatus; and the rather stout platinum wire, which extends from *f* to *j*. The porcelain tube *c* is joined by rubber tubing to the branched tube *d*. The latter is fitted with a stopper *g*, through which passes the stout platinum wire *e*, which extends into the porcelain tube to the point *h*, where it is joined to a thinner platinum wire. This wire, extending from *h* to *j*, has a length of 1·75 metres, and weighs about 2·5 grams. It passes through the porcelain tube, and then returns outside in a series of loose coils. The thicker wire at the ends is necessary to avoid overheating of the rubber stoppers. A roll of copper wire gauze (*b*), about 60 mms. in length, is inserted between the boat and the porcelain tube. This last is made (by the Royal Berlin Porcelain Works) of unglazed porcelain. Clay-pipe stems may also be used, but are very hygroscopic, and require careful burning out before each combustion. In making a determination, oxygen or air is admitted at *d*, and a gradually increasing current is sent through the platinum wire. The maximum current used amounts to about 3·6 ampères at 54 volts. At the same time, the roll of copper gauze and the boat are heated by a lamp held in the hand. The part of the glass tube into which the porcelain tube extends is protected by an asbestos trough below and an inverted mica trough above. It is also useful to invert an asbestos trough some little distance above the copper gauze. During the cooling the current should be shut off very gradually, and it is advisable to protect the exposed parts of the combustion-tube by covering them with soot from a smoky flame. Care must also be taken that none of the platinum loops touch the glass at any point, as this leads to cracks.

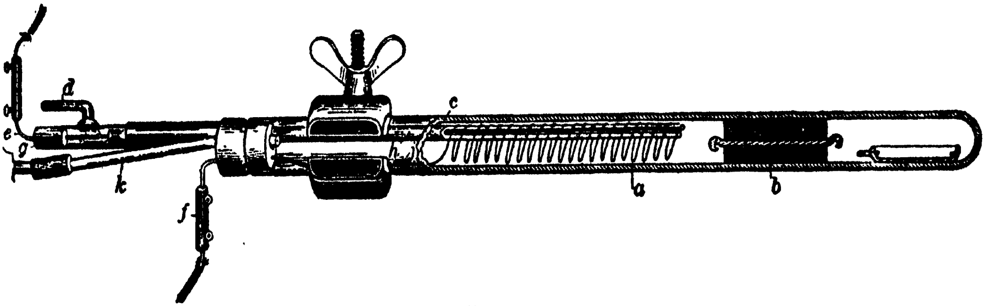


FIG. 1.

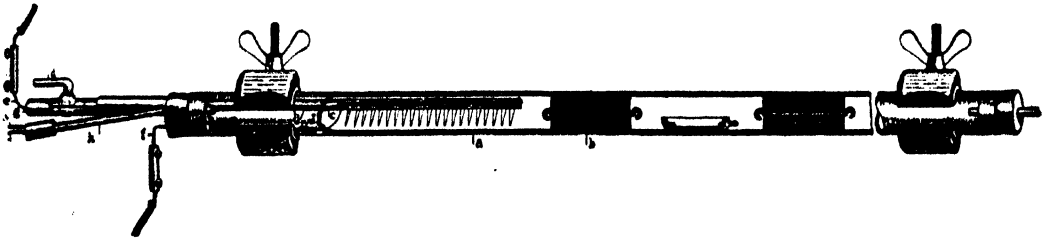


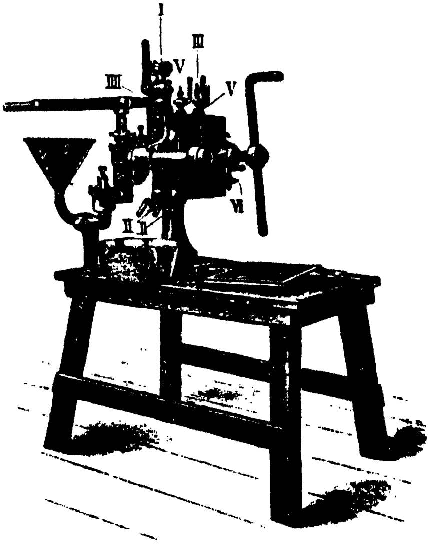
FIG. 2.

The objections to this form of the apparatus are that the whole of the heating apparatus, as well as the copper gauze, must be removed before each combustion, and that it requires a little time to sweep out the products of combustion from the closed end. A tube open at both ends is therefore to be preferred, and is shown in Fig. 2. The procedure is practically the same as before, except that a current of air or oxygen is admitted from the rear of the tube as well as at *d* throughout the entire combustion, and that an additional roll of copper gauze, heated by a second lamp, is placed behind the boat. For substances containing halogens, nitrogen, or sulphur, the usual modifications in the packing of the tube are made. The authors use lead chromate in the form of a cartridge made by filling a shell of fine copper gauze with granular lead chromate.

A large number of excellent test analyses made with the apparatus described above are given. For substances containing carbon and hydrogen, or carbon, hydrogen, and oxygen only, the time required was usually only thirty minutes or less. Halogen and sulphur compounds also burnt readily, but with nitrogen compounds it was found wise to allow about one and a half hours for the combustion.

A. G. L.

A New Filtering Apparatus. (*Chem. Zeit.*, 1905, xxix., 511.)—The firm of Ernst. W. Hübner, of Ammendorf b. Halle a. S., have introduced a small apparatus, constructed on somewhat similar lines to their larger filter-presses, for filtering.



As some substances can be filtered only under the natural pressure given by a column of water a few metres in height, others can at once have pressure applied to them by means of a special pressure-pump. There are also cases where it is desirable to commence filtering under natural pressure, and afterwards to apply pressure. The apparatus under notice is specially designed to meet the last-mentioned cases, but it can be equally well used for filtering either with or without additional pressure. There is also an arrangement by which the liquid to be filtered can be cooled or heated to any temperature desired by means of a current of water or steam.

All this is attained in a fairly simple manner by the proper arrangement of pipes and taps as shown in the figure. Tap I. is connected with a reservoir of water, when open, filtration proceeds at the pressure of a column of water the height of the reservoir; when the tap is closed pressure by means of the pump can be applied. Taps II. II. provide for the egress of the filtrate, tap III. for the addition of wash-water, IIII. for the admission of air. Taps V. V. and VI. can be connected either with a steam or a cold water supply.

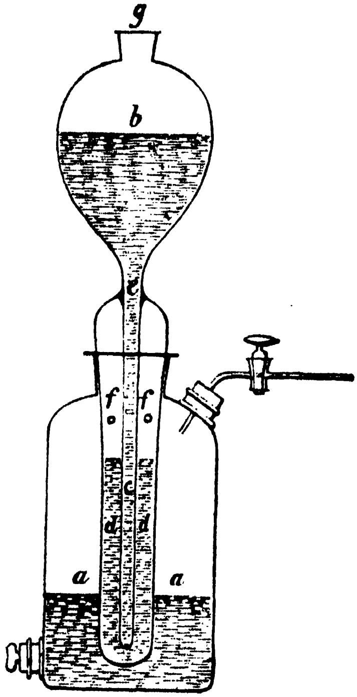
E. K. H.

On a Difficulty in Dissolving the Fused Mass out of Platinum Crucibles. C. Bender. (*Zeits. angew. Chem.*, 1905, 1025.)—The author recommends that all platinum crucibles should be polished with sea-sand between every few, at mos four, alkaline fusions.

Failure to do so results in the formation of a rough surface, from which it is difficult to dissolve off the fused mass.

The irregular shape of crucibles in constant use show, that in many laboratories this simple precaution is often neglected. A. N. C.

A New Apparatus for the Supply of Gases. Emil Geisel. (*Chem. Zeit.*, 1905, xxix., 726.)—The problem of obtaining a steady and continuous supply of various gases, especially hydrochloric acid and sulphur dioxide, is one entirely of the apparatus to be used.



The author refers to two previous attempts to solve the question: First, that of Bender, which apparatus the author considers defective in detail, and high in price, owing to its requiring a large amount of skilled glass-working in its construction; second, that of McCoy, which has the objection that it has a rubber cork as an essential part of its interior fitting, which would come into contact with the sulphuric acid used in both the important cases mentioned above.

The apparatus designed by the author is of fairly simple construction, and the method in which it works can be clearly seen from the accompanying figure.

The vessel *a* contains one of the liquids used for obtaining the gas, in the case of sulphur dioxide a concentrated solution of sodium bisulphite. In *b* is concentrated sulphuric acid. In action, the acid passes down the stem *c*, up the outer jacket *d*, and through the four fine holes *f* (two only shown in section). Thus a very steady current of gas can be obtained.

There is a stopper at the base of *a* by which *d* can be emptied and cleaned, the acid being retained meanwhile in *b* by inserting a well-fitting cork in *g*.

The apparatus can be used for almost any gas, and the author recommends it as especially convenient for the production of a steady stream of air-free carbon dioxide in nitrogen estimations—in this case *a* should contain solid sodium bicarbonate as well as a saturated boiled solution of it, whilst *b* is filled with boiled hydrochloric acid.

The apparatus is supplied by the firm of Victor Lietzau, Danzig. E. K. H.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE COMPOSITION AND ANALYSIS OF MILK.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, June* 7, 1905.)

Average Composition of Milk.—Of the 38,297 samples analysed in the laboratory of the Aylesbury Dairy Company during 1904, 33,747 were samples of milk. The average composition of 15,910 samples taken from the milk on arrival at the depots from the farms is given in the subjoined table :

Average Composition of Milk during 1904.

Month.	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January	1·0324	12·58	3·61	8·97	1·0322	12·96	3·98	8·98	1·0323	12·77	3·80	8·97
February	1·0327	12·54	3·57	8·97	1·0322	12·86	3·89	8·96	1·0325	12·70	3·73	8·97
March	1·0325	12·52	3·55	8·97	1·0322	12·82	3·86	8·96	1·0323	12·67	3·71	8·96
April	1·0323	12·36	3·45	8·91	1·0320	12·81	3·91	8·90	1·0321	12·58	3·68	8·90
May	1·0327	12·24	3·28	8·96	1·0323	12·85	3·87	8·98	1·0325	12·54	3·57	8·97
June	1·0327	12·28	3·29	8·96	1·0322	12·75	3·81	8·94	1·0323	12·50	3·55	8·95
July	1·0321	12·26	3·42	8·84	1·0314	12·64	3·87	8·77	1·0317	12·45	3·64	8·81
August	1·0321	12·33	3·48	8·85	1·0315	12·77	3·96	8·81	1·0318	12·55	3·72	8·83
September	1·0324	12·50	3·53	8·96	1·0320	12·96	4·03	8·93	1·0322	12·73	3·79	8·94
October	1·0324	12·60	3·63	8·97	1·0320	13·07	4·11	8·96	1·0322	12·84	3·87	8·97
November	1·0326	12·72	3·70	9·02	1·0321	13·09	4·11	8·98	1·0324	12·90	3·90	9·00
December	1·0327	12·76	3·71	9·05	1·0323	13·08	4·07	9·01	1·0325	12·92	3·89	9·03
Average	1·0325	12·47	3·52	8·96	1·0320	12·89	3·96	8·93	1·0322	12·68	3·74	8·94

The morning and evening meals have been kept separate, and, as usual, there is a difference of about 0·4 per cent. of fat between them. As in former years, May and June are the poorest months, and October, November, and December the richest

The yearly average percentage of fat (3·74) is less than that found last year; it may be suggested that the very wet season of 1903 affected the soil, and the fodder grown on the farms in 1904 was not of the same quality as in the years immediately preceding, and that this had some effect on the percentage of fat in the milk.

Human Milk.—A sample of human milk gave the following figures:

	Per Cent.
Total solids	10·39
Fat	1·98
Sugar	6·40
Proteids	1·75
Ash	0·26
Solids-not-fat	8·41

The fat in this sample is very distinctly below the average.

Chlorine in Ash.—In order to see whether much chlorine was lost on burning milk, as has been shown by J. O'Sullivan to be the case in vegetable ash, a sample giving 0·092 per cent. chlorine in the ash was burnt with lime; 0·100 per cent. was found, showing that 0·008 per cent. had been lost.

Buttermilk.—Three methods of preparing buttermilk may be used:

Churning soured cream;
Churning sweet cream;
Churning soured milk.

A preparation much resembling buttermilk may be also made by churning sour separated milk till the curd is broken up into very fine particles.

The composition of each of these is:

	Sour Cream.	Sweet Cream.	Milk.	Separated Milk.
Specific gravity	1·0314	1·0331	1·0329	1·0355
Total solids	8·39	9·02	8·87	9·23
Fat	0·50	0·35	0·70	0·10
Sugar	3·40	4·42	3·65	3·93
Proteids	3·30	3·51	3·28	3·65
Ash	0·65	0·73	0·68	0·79
Lactic acid	0·50	0·01	0·76	0·56

A slight addition of water for churning purposes was made to the three buttermilks.

A New Milk Preservative.—Quite recently a new preservative came into my hands; it consisted of a liquid of specific gravity 1·027 containing 5·6 per cent. of hydrogen peroxide, some sodium chloride, and a little sodium phosphate, and pellets chiefly consisting of potassium carbonate (with another potassium salt which, owing to the sample being accidentally destroyed before the analysis was finished, was not identified). Organic matter was also present.

In the circular accompanying the sample it was claimed that this preservative was harmless, contained nothing but constituents of milk, and could not be detected. As hydrogen peroxide is not immediately destroyed when added to milk, but remains for some time, it would probably be found in samples to which this preservative had been added. The test with para-phenylenediamine or "ortol" will detect very minute quantities.

The Gerber Method.—There appears to be some doubt as to the correct volume of one division of the neck of the Gerber bottle, and not only do the bottles of different makers vary slightly in the volume of the divisions, but the English and German standardizing authorities are not quite in accord as to the value. I have found that the value 0·126 c.c. for each division, indicating 1 per cent. of fat, gives the most accurate results, and that when bottles graduated with a smaller value were used the readings required to be corrected to this volume.

As values of from 0·124 to 0·126 appear to be used by the makers of bottles, I have made some fresh experiments with a view to determine (*a*) the most nearly correct value, (*b*) the errors inherent to the process, and (*c*) the influence of diameter of the neck on the results.

A series of milks were prepared by weighing out quantities of cream and separated milk and mixing them, and the fat in each of these was determined by the Gerber process and the results compared with the percentage of fat deduced from the composition of the cream and separated milk. The mean of four Gerber readings by Messrs. Goodson and Ashby and myself was taken, and only in one milk did the extreme readings differ by as much as 0·1 per cent. A set of bottles graduated, using the value 0·124 c.c., was used, and in order to minimize errors due to imperfect graduation each milk was read in four different bottles.

A series of determinations of the weight of milk delivered by an 11 c.c. pipette, the three observers agreeing very closely, gave the value as 11·276 grams − 0·017 F.; with ordinary milks (3 to 4 per cent. fat) 11·22 grams is very nearly correct.

I estimate the volume of the fat between the upper and lower layers of the meniscus as equal to 0·08 per cent., and this, which is always neglected in reading the percentage of fat, must be added on in calculating the actual volumes.

The volumes of fat are calculated by multiplying the percentage of fat by the weight of milk calculated from the formula given above, and dividing by 100 × 0·8865 (the mean apparent density of fat in glass at 70° C.).

The following table shows the results obtained:

Percentage of Fat.	Mean Gerber Reading.	Mean Gerber Reading $\times \frac{126}{124}$.	Weight of Milk delivered. Grams.	Volume of Fat at 70°. C.c.	Volume of Fat in Gerber. C.c.	Ratio.
1·37	1·38	1·36	11·253	0·174	0·181	1·04
1·84	1·88	1·85	11·245	0·233	0·243	1·04
2·76	2·79	2·74	11·229	0·347	0·356	1·03
3·23	3·29	3·23	11·221	0·409	0·418	1·02
3·66	3·74	4·68	11·217	0·463	0·474	1·02
4·45	4·54	3·46	11·200	0·562	0·573	1·02
4·92	5·04	4·96	11·192	0·621	0·635	1·02

It is seen that the value 0·126 c.c. for one scale division represents very closely the percentage of fat in milk, and that the volume of the Gerber fat is greater than the actual fat present, the mean ratio being 1·025.

There are several reasons which might explain the increased volume: (*a*) There is a layer of acid liquid formed round the fat; (*b*) amyl alcohol, or a derivative, dissolves in the fat; and (*c*) the acid and amyl alcohol produce amyl esters, with elimination of glycerol, or other changes.

If (*a*) is the reason, a marked difference in the volume should be observed in bottles with necks of different diameters; a bottle of diameter 0·312 cm. gave a volume 0·177 c.c., a bottle of diameter 0·334 cm. gave a volume 0·176 c.c., and a bottle of diameter 0·496 cm. gave a volume 0·175 c.c.; the differences represent about 0·01 in the reading, which is within the limits of experimental error.

As surface energy to which a layer would be due varies greatly with temperature, the apparent expansion of the fat should be markedly different from the true expansion in the presence of a layer; on cooling a fat column 7·45 divisions from 76° to 34·5°, it contracted to 7·20, a difference of 0·25 divisions, and on cooling a fat column 7·19 divisions from 74° to 30° it contracted to 6·96, a difference of 0·23 divisions. A contraction of 0·24 division should have taken place in each case. These results show that if any layer is formed it is of infinitesimal thickness, and the increase in volume cannot be due to this cause.

If (*b*) is the reason, a variation of the amount of amyl alcohol should affect the results; using quantities from 0·8 c.c. to 2·0 c.c. in place of the usual 1·0 c.c. produced no appreciable variation in results. Therefore the increase in volume cannot be due to amyl alcohol or a derivative dissolving in the fat.

An examination of the fat showed that the real cause is a change in the composition of the fat. The same milk was treated forty-eight times, and the combined fat separated. Some of the fat was churned from the cream obtained direct from the milk.

14·666 grams of the Gerber fat were steam-distilled, and a marked pear-drop odour was noticed in the distillate. The acidity was estimated both in the distillate and the aqueous residue in the distilling-flask, and the sulphuric acid was also estimated as barium sulphate in the aqueous residue. The aqueous residue was turbid, and after saponification and acidification some solid fatty acid separated. The distillate was saponified, and the alkaline solution steam-distilled; oily drops came over, which on oxidation with chromic acid gave a strong valeric acid odour. The Gerber fat, the fat which had been steam-distilled, and the original milk fat were examined. The results were:

	Gerber Fat.	Steam-distilled Fat.	Original Fat.
Total acidity (as c.c. N acid per 100 grams)	25·2	17·0	0·5
Volatile acidity (do.)	2·2	—	—
Water soluble acidity (do.)	5·9	—	—
Of which H_2SO_4	4·1	—	—
Volatile acids (as butyric)	5·42%	4·98%	5·87%
KOH absorption	22·04%	21·92%	22·68%
Glycerol	10·49%	—	11·95%
Zeiss refraction figure at 35° C.	42·5°	45·1°	47·1°
Apparent density in glass at 70°	0·881	—	—
H_2SO_4 in fat	—	0·20	—

From these results it appears that the fat has increased in weight about 3 per cent., and has lost 0·3 per cent. of butyric (or other soluble) acid; about 6 per cent. of the glycerides have been converted into free acid, about 18 per cent. into amyl esters, and about 40 per cent. into diglycerides or the equivalent of monoglyceride; the fat has taken up 0·39 per cent. of sulphuric acid, probably as sulpho-oleic acid; its volume has increased, owing, probably, to the amyl esters having a greater specific volume than glycerides, about 0·6 per cent. Altogether the total increase in volume is rather over 3 per cent., a figure which is in good agreement with the increase (2·5 per cent.) calculated above. Whether the difference is a real one, due to a slight solubility of fat in the sulphuric acid amyl alcohol solution (? as sulpho-oleic acid), or to the churned fat not being quite of the same composition as the ether-extracted fat, or due to experimental error, is a matter for speculation. The difference is not enough to affect the conclusion that the increased volume of the fat is due to a change in the composition of the fat.

I conclude, therefore—

(*a*) The best value for the scale division of a Gerber bottle is 0·126 c.c.

(*b*) The volume of the fat obtained in the Gerber process is about 1·025 the volume of the fat in the milk.

(*c*) The neglect of the volume of the fat between the upper and lower parts of the meniscus is partly compensated by the decrease of the weight of milk delivered by an 11 c.c. pipette with increasing quantities of fat.

(*d*) The exact diameter of the neck of the bottle is of no importance.

The results obtained show that the method devised by Rosier and myself (ANALYST, xxiv., 172) is accurate only to the extent of the compensation of two opposing factors—the increase in the weight of fat, and the decrease owing to solubility and volatility of some of the products formed. These two appear, however, to balance each other with fair accuracy.

DISCUSSION.

Mr. E. R. BOLTON inquired whether the iodine absorption of the fat obtained in the Gerber process had been determined.

Mr. RICHMOND replied in the negative, but said that he should expect the iodine absorption of the butter-fat to be lowered.

Mr. A. C. WILSON asked whether Mr. Richmond could give any information as to the difference in quantity between the morning and evening yields in these cases. In the North Riding of Yorkshire and the county of Durham, where the milking intervals were very uneven, the morning milk was practically speaking one-third more in quantity than the evening milk.

The PRESIDENT (Mr. Bevan) asked whether the decrease shown during 1904 in the average percentage of fat was due to any unusual circumstances connected with the process of analysis or to actual deterioration in the quality of the milk.

Mr. RICHMOND said that, according to an average which he had some time previously worked out for the samples which came under his observation, the interval between the morning and the evening milkings was 10·8 hours. The ratio between the quantities yielded in the morning and in the evening was approximately 100 : 85. With regard to the President's question, he thought that, seeing that the analyses were all made in the same manner and by the same operators, that the apparatus and reagents were constantly checked, and that the number of analyses was about 15,000 each year, the experimental error of the average could not be very large. He therefore considered that there must be some real difference in the milk itself. He believed that there was, and would be, a constant tendency for the percentage of fat in milk to fall, owing to the practice which prevailed among farmers of weeding out all cows giving small quantities of milk. This, in his experience, meant that those cows were being got rid of which gave the highest percentage of fat. The slight increase in the average percentage of fat shown during the three previous years he was inclined to attribute to increased attention to quality on the part of the farmers following the issue of the Sale of Milk Regulations. Probably the process of breeding for quantity was again coming to the fore. There was, however, one special circumstance in 1903, namely, the abnormal rainfall of that year. Possibly that had some influence which was reflected in the milk of 1904.

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A SIMPLE AND CONVENIENT CAMERA FOR PHOTO-MICROGRAPHIC WORK.

By A. C. WILSON, F.R.S.E.

(*Read at the Meeting, June* 7, 1905.)

Those who find the camera to be a necessary accessory to the microscope as a ready means of recording their observations will find the simple arrangement which is shown in the two subjoined figures convenient and efficient for general laboratory work.

The arrangement of the apparatus aims at simplicity, freedom from vibration, and portability.

The camera is made of sheet-brass, and is 10 inches long and 4 by 4 inches, taking a 3¼-inch plate. It is fixed upon an adaptor, which runs between two parallel guides, and may be clamped in any position by means of a thumb-screw. If a larger picture is required, a half-plate camera of similar construction to the above is substituted, in which case the adaptor is not necessary.

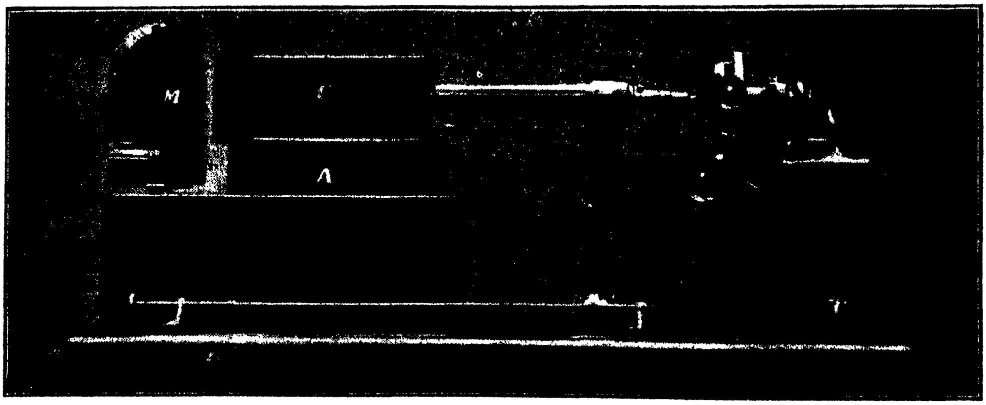

Fig. 1.
C, camera ; M, mirror ; T, revolving table ; B, base board ; A, adaptor.

The microscope is fixed upon the revolving table and may be used in any position. The focussing is accomplished by means of a mirror, which may be moved to any angle, thus enabling the worker to make his observations, and if necessary take the photo-micrograph, without moving from his position in front of the instrument.

The whole arrangement may be worked either in a vertical or a horizontal position; but in the former case a suitable stand is used having rigid supports, to which the base carrying the camera and microscope is attached by means of a thumbscrew.

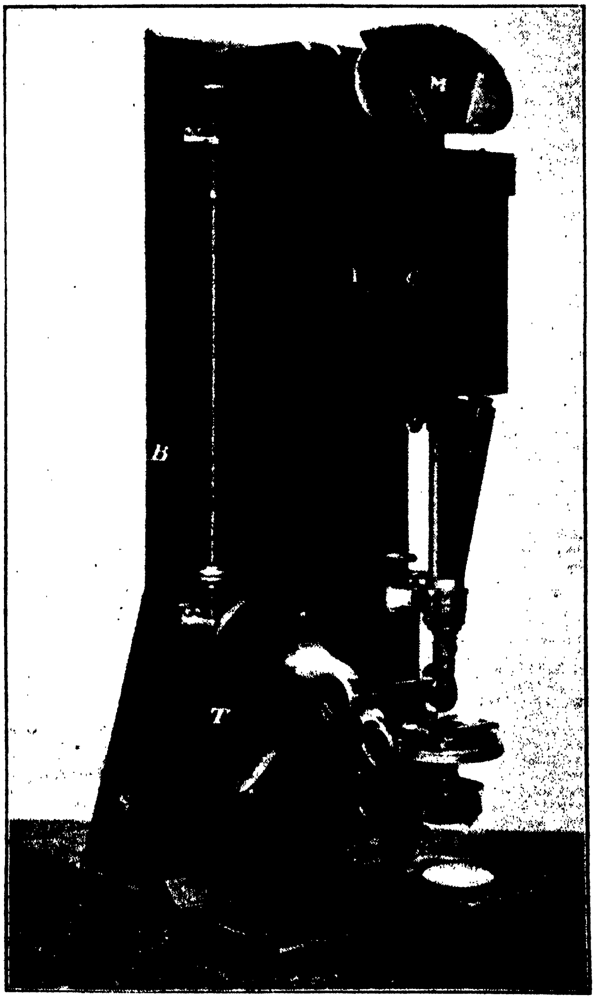

Fig. 2.

C, camera ; M, mirror ; T, revolving table ; S, stand ; B, base board ; A, adaptor.

The camera being of a fixed length, every photo-micrograph is of the same size if taken with the same combination of lenses.

Discussion.

The President (Mr. Bevan), in inviting discussion, said that, in his experience, which, however, had been almost entirely confined to the use of a very long camera

—about 5 feet long—the photographs taken without the eye-piece were as a rule sharper and clearer and the field was flatter. He thought the idea of the mirror an excellent one.

Mr. J. H. B. JENKINS said that Mr. Riddick and he had experimented with a mirror to reflect the image, much as in the author's apparatus; but they had found that, with the full-plate camera they used, the arrangement was inconvenient, and they had discarded it. Nearly the whole of their work had been done with opaque objects; but they had had some experience in the photo-micrography of starches, and had found it best in that case to use a vertical camera. If a horizontal camera were used the microscope slide would be vertical, and the starch, being mounted in a fluid medium, took a long time to settle down, which was not the case with a vertical camera and a horizontal slide.

Mr. RICHMOND said he understood that the object of using the mirror was to do away, partly if not entirely, with the necessity for the focussing-rod. He noticed that the apparatus shown had both a focussing-rod and a mirror, and he should like to hear under what circumstances the one or the other would be used. He also drew attention to the device of cementing one or two thin cover-glasses on to the ground-glass screen with Canada balsam. A general idea of the image was obtained on the screen, and the fine adjustment with a lens was performed on the clear spaces.

Mr. WILSON said that if a high power was being used, and the focussing done with the aid of an eye-piece or other suitable lens upon a plane-glass screen or a plate of extremely fine ground glass, it was sometimes desirable to remove the mirror and use the rod in the final adjustment, especially in cases where the illumination was difficult. As to whether it was best to use an eye-piece or not depended upon the apparatus at the disposal of the worker and the nature of the object under examination. Without the eye-piece, and by using a sufficiently long camera, it was of course possible to obtain good results, but not, he thought, better than could be obtained by a suitable combination of lenses. The short camera had many advantages, and the movable mirror was a great convenience. He had also found it a good plan to use a ground-glass screen for focussing, and to cement one or two very thin cover-glasses on to it with Canada balsam. The screen was thus made transparent at those places, so that the fine adjustment could be made there with a lens after the general outline had been focussed on the ground glass.

Mr. JENKINS said that the application of a little oil produced much the same result as cementing on a cover-glass.

Mr. CHAPMAN said that in his experience a better definition was obtained with a projection eye-piece than with an ordinary one, though at the sacrifice of a certain extent of field.

The PRESIDENT said that his practice was to focus roughly on a ground-glass screen, and then substitute a sheet of plain glass, the inside of which was allowed to become dusty, the final adjustment being made by focussing on the dust with a lens. In that way the focus was obtained in the exact position occupied by the sensitive film.

Mr. WILSON said that, unlike Mr. Chapman, he personally preferred an ordinary to a projection eye-piece; but a good deal depended on the nature of the work and on the worker's knowledge of the instrument used. The greater portion of his own work involved no higher magnification than about 300 diameters.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Chemical Detection of Rancidity in Butter. P. Soltsien. (*Chem. Rev. Fett-u. Harz Ind.*, 1905, xii., 177, 178.)—The products causing the characteristic effects of rancidity can be distilled in a current of steam, and if the distillate is treated with alkali in excess to neutralize the fatty acids, and again distilled, they are obtained in a more concentrated form. The products from rancid lard give strong aldehydic reactions, such as the reduction of ammoniacal silver nitrate, but these hardly occur in the case of the distillate from rancid butter. Welman's phosphoric acid reagent is recommended as a reliable chemical test. It can be applied directly to a colourless fat, and on treating the mixture with ammonia in excess, a more or less pronounced blue coloration is produced, according to the degree of rancidity. In the case of a coloured oil or fat, such as butter, the test is applied to the distillate obtained as described above, and this should also be done whenever any green coloration is produced on the direct addition of the reagent to a colourless fat. The reaction with ammonia does not take place until after thirty seconds to a minute. The test can be carried out simultaneously with a determination of the free volatile fatty acids. C. A. M.

Detection of Wood Spirit in Essences, Brandies, Tinctures, and Fluid Extracts. R. Peters. (*Chem. Zeit. Rep.*, 1905, xxix., 218.)—The wood spirit used to denature brandy for commercial purposes must contain 25 per cent. acetone. This fact makes the detection of wood spirit in brandy, etc., very easy. The author considers Légal's process the best; this is carried out as follows: 10 c.c. of the alcohol-containing material are mixed with water, and 20 c.c. of the mixture distilled off, using a small flame. To 10 c.c. of the well-mixed distillate there is added 1 c.c. of a freshly prepared 1 per cent. solution of sodium nitro-prusside (this must be kept from the light), and then 2 c.c. of an approximately 4 per cent. solution of caustic soda; a greater quantity of the latter must be used for strongly acid liquids. If acetone is present, a more or less deep red colour appears; in the absence of acetone, only a yellow colour is obtained. Colourless preparations—*e.g.*, spirit of camphor, etc.—can be tested without previous distillation. The author hopes to succeed in modifying Légal's, or one of the other methods, so as to make it quantitative. E. K. H.

The Determination of Glycerin in Wines. X. Rocques. (*Ann. de Chim. anal.*, 1905, x., 306-309.)—This is a method of direct determination based on the solubility of glycerin in a mixture of acetic ether and alcohol. Acetic ether by itself will dissolve only 13·4 grams of glycerin per litre, but a mixture of 80 per cent. of acetic ether with 20 per cent. of pure alcohol dissolves 80·6 grams per litre at 20° C., without dissolving any appreciable quantity of other substances in the wine. In the determination 200 c.c. of the wine are evaporated to a syrup, and the residue mixed with a quantity of quicklime equal to that of the sugar present. The pulverized mass is allowed to stand twenty to thirty minutes, and then triturated with 50 to 200 c.c. of alcohol (96 to 97 per cent.), added little by little, until a homogeneous creamy fluid is obtained. This is allowed to stand for twenty to thirty minutes, and then filtered from the insoluble calcium sucrates, the filter being subsequently washed with alcohol. The filtrate is rendered acid with tartaric acid, and the bulk of the alcohol distilled, a little pumice-stone being added to prevent bumping. The residue (15 to 20 c.c.) is mixed with 3 to 5 grams of quicklime and 10 grams of fine sand in a flat dish, and the evaporation completed *in vacuo*, which takes about twelve hours. The dry powder is shaken for two hours in a well-closed flask with 100 c.c. of the above-mentioned mixture of acetic ether and alcohol, after which it is allowed to stand, and then filtered. An aliquot portion of the filtrate (say 75 c.c.) is placed in a weighed dish, and evaporated *in vacuo* over sulphuric acid, and the residue of glycerin left for three days in the vacuum before being weighed. It will be found quite free from sugars and to contain only traces of mineral matter.

C. A. M.

A New Method of Estimating the Aldehydes in Oil of Lemon. E. Berté. (*Chem. Zeit.*, 1905, xxix., 805.)—The author refers to the controversy which took place in the *Chemist and Druggist* in the autumn of 1903, in which most of the authors were of opinion that the percentage of citral did not exceed 3 to 4 per cent.

Sadtler, however, used a process founded on an observation of F. Tiemann, in which a dilute aqueous solution of neutral sodium sulphite is shaken up with the aldehyde-containing liquid. Citral, under these circumstances, is transformed into citral dihydro-sulphonic acid, and the equivalent quantity of sodium hydroxide is liberated. Citronnellal does not give such a reaction. Tiemann found, by this method, 5·26 per cent. of citral; the total amount of aldehydes must be greater.

The author, whilst having complete confidence in the bisulphite method, as proposed by himself and Soldaini (*Chemist and Druggist*, September, 1903), has worked out a simple method for the estimation which depends on the difference in optical activity of the oil before and after treatment with one of the general reagents. The process is carried out as follows: The rotation due to the original oil was first determined. Ten c.c. of the oil and 50 c.c. of a saturated solution of potassium bisulphite were placed in a 250 c.c. Erlenmeyer flask; the flask was closed by a cork provided with one hole, through which passed a glass tube 40 to 45 centimetres in length. The mixture was shaken to an emulsion, kept on a boiling water-bath for ten minutes, and repeatedly shaken. It was allowed to cool, again heated for five minutes with vigorous shaking, and then again allowed to cool. The mixture was then transferred to a separating funnel of 100 c.c. capacity; it was allowed to stand some time for the upper layer of oil to separate from the lower layer containing the combined aldehyde. The separated terpene was twice washed with a small quantity of distilled water, separated, dried by addition of a small amount of anhydrous sodium sulphate, and filtered. The clear liquid was then examined polarimetrically. The percentage of aldehyde is given by the formula—

$$C = \frac{100 (A - a)}{A}$$

where a is the rotation of the original oil, and A that of the oil after removal of the aldehyde. The author performed several series of experiments to test the method. He first tried whether the rotatory power of the terpene was affected by the action of the ordinary aldehyde reagents or by heat. These experiments were performed with a terpene of specific rotation +66° at 20° C., and specific gravity 0·85190 at 15° C. No change could be observed.

Some experiments were next performed on genuine oil of lemon, obtained by favour of the producers. Different samples gave values of 6·85 to 7·40 per cent. of aldehyde.

Another series of determinations was made on lemon oils adulterated with known quantities of a specially prepared terpene, or mixtures of this terpene and olive oil such as to give a specific gravity about that of the unadulterated oil. The oil was first analysed to prove its purity. The figures obtained agreed fairly with the calculated ones.

Experiments were then made on mixtures of citral and limonene of known composition. The citral used was a pure citral from lemon-grass oil, optically inactive, made by Kahlbaum; the limonene was obtained from genuine oil of lemon, and had a specific rotation of +66° at 20° C. The figures obtained are worth giving in detail:

Quantity of Limonene used.	Quantity of Citral added.	$[\alpha]_D$ at 20° C. of the Mixture.	$[\alpha]_D$ at 20° C. of the Terpene obtained by Treatment with Potassium Bisulphite.	Difference.	Quantity of Citral.	
					Found Per Cent.	Calculated Per Cent.
19·8	0·2	+65° 28′	+66° 0′	+0° 72′	1·10	1·00
19·6	0·4	+64° 70′	+66° 0′	+1° 30′	1·97	2·00
19·4	0·6	+64° 0′	+66° 0′	+2° 0′	3·04	3·00
19·2	0·8	+63° 30′	+66° 0′	+2° 70′	4·10	4·00
19·0	1·0	+62° 70′	+66° 0′	+3° 30′	5·00	5·00
18·8	1·2	+62° 0′	+66° 0′	+4° 0′	6·07	6·00
18·6	1·4	+61° 30′	+66° 0′	+4° 70′	7·13	7·00
18·4	1·6	+60° 70′	+66° 0′	+5° 30′	8·04	8·00
18·2	1·8	+60° 0′	+66° 0′	+6° 0′	9·10	9·00
18·0	2·0	+59° 30′	+66° 0′	+6° 70′	10·16	10·00

Finally, observations were made on a terpene-free "concentrate," for which the author wishes to thank Messrs. Santi de Pasquale. This gave the following figures:

Specific rotation at 15° C.	9·20°
Specific gravity	0·8975
Quantity of ester (as geranyl acetate)	19·55 per cent.
Refractive index at 15° C. (Zeiss refractometer) ...	89

The process detailed above was then applied, and a difference in specific rotation of −13·1° was obtained. Knowing the quantity of geraniol present from the first analysis, the quantity of citral could be calculated, and was 58·49 per cent. by volume, or 65·72 per cent. by weight.

The author is pursuing investigations of other terpene-free concentrates, in order to establish the ordinary amount of aldehyde therein. E. K. H.

A Reaction of Laurel Oil. Monpurgo. (*Giorn. di Farm. di Trieste*, 1905, 353; *Ann. de Chim. anal.*, 1905, x., 327.)—Laurel oil is stated to be frequently adulterated. The pure oil, when shaken with twice its volume of alcohol and filtered, yields a green filtrate the colour of which is not modified by the addition of ammonia, but is changed to yellowish-green by hydrochloric acid. The iodine value ranges from 66 to 78, the saponification value from 197 to 210, and the melting-point from 32° to 36° C. When the oil is heated with twice its volume of alcohol and the liquid cooled slowly the white mass that separates appears amorphous under the microscope. When several drops of an ethereal solution of the oil are thrown into absolute alcohol the mixture remains clear in the case of a pure product. C. A. M.

Note on the Analysis of the Compound Tincture of Camphor (Paregoric). F. C. J. Bird. (*Pharm. Journ.*, 1905, lxxv., 154.)—As no analytical method is described or suggested in the British Pharmacopœia for determining the strength in morphine of a sample of this tincture, the official process for tincture of opium might be considered as applicable. However, to work with the equivalent of the official quantity of 80 c.c. of tincture of opium, it is necessary to employ no less than 1,300 c.c. of compound tincture of camphor. Fair results may be obtained by taking only 250 c.c., but with smaller quantities than this the amount of morphine found is too low. If only 10 c.c. of the sample be assayed, the alkaloid may be missed altogether.

The following process answers well with as little as 2·5 c.c. of the tincture, although 10 c.c. is recommended as the most suitable quantity: 10 c.c. of the sample are evaporated to dryness on a water-bath, the residue is taken up with dilute alcohol and a minute drop of acetic acid, again evaporated, and finally dissolved in 2 c.c. of water. The solution is poured through a filter, the filter is washed, and the filtrate and washings extracted three times with hot amyl alcohol, after adding a little potassium carbonate. The united extracts are evaporated to dryness. The residue is then dissolved in a little dilute hydrochloric acid, filtered through a small filter containing charcoal, and washed. The clear solution is now rendered alkaline by the addition of a little powdered ammonium carbonate, and extracted three times with hot amyl alcohol. The mixed extracts should measure about 8 c.c., and are evaporated to dryness. The usual reactions may be applied to portions of a solution of this final residue, and the colorations obtained compared with those yielded from 10 c.c. of a sample of compound tincture of camphor of known strength.

W. P. S.

The So-called "Manipulated" Opiums. P. Guigues. (*Journ. Pharm. Chim.*, 1905, xxii., 103-107.)—According to the author, Smyrna opium normally contains from 12 to 14 per cent. of morphine, though in some cases, owing to part of the pericarp being removed with the coagulated latex, it sometimes contains less. Such natural, though inferior, products, are exported to Germany, England, and the United States, where the Pharmacopœias require opium to contain about 10 per cent. of morphine. But, with this exception, it is stated that all opiums containing a low percentage of morphine are products "manipulated" in Smyrna for French and Italian consumption; the amount being reduced to 10 per cent. for France and to 6 per cent. for Italy. The author has received samples and details of the method of preparation from Smyrna dealers. The fresh opium is converted into paste and mixed with apricot pulp or similar products, so as to reduce the proportion of morphine to the required extent, after which the mass is divided into small cakes, which are wrapped round with the fruits of a species of rumex. The analysis of three samples gave the following results:

Sold as—	Water. Per Cent.	Ash. Per Cent.	Aqueous Extract. Per Cent.	Morphine. Per Cent.	Narcotine. Per Cent.
No. 1. Opium, superior quality	11·45	4·10	46·48	11·18	1·65
No. 2. ,, average quality	12·12	3·60	55·98	nil.	2·37
No. 3. ,, inferior quality	10·30	6·15	29·78	1·9	1·29

All the results were calculated on the dry substance. The extract of No. 1 and No. 2 had the normal red-brown colour, while that of No. 3, which swelled up into a gelatinous mass, was brownish-yellow. No. 1 was forwarded as a genuine opium, whilst No. 2 was stated to have been mixed with pulp, and No. 3 with coagulated white of egg. The amount of reducing sugars (as dextrose) in No. 2 was 10·25 per cent., while No. 1 gave 8·6 per cent. and No. 3 2·85 per cent. Hence, assuming that No. 1 was really a pure product, the amount of reducing sugars is not conclusive of adulteration. Sample No. 3 contained a large amount of nitrogen (*cf.* ANALYST, xxx., 310).

C. A. M.

The Determination of Morphine in Opium. L. Valenti. (*Giorn. di Farm. di Trieste*, 1905, 289; *Ann. de Chim. anal.*, 1905, x., 326.)—From the results of comparative determinations by different methods the author recommends that of Fluckiger (precipitation from a solution in ether-alcohol). The only drawback is the solubility of the morphine in the solvent. This amounts to 0·118 gram per 100 c.c., and ought to be added to the weight of morphine found.

C. A. M.

Meconic Acid in the United States Pharmacopœia, 1890, Assay of Opium and Certain Meconates. Edward Mallinckrodt, jun., and Edward A. Dunlap. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 946.)—In making an assay of opium by the United States Pharmacopœia (1890), process, in which the morphine is precipitated from the aqueous extract by dilute ammonia after the addition of some alcohol and ether, the formation of a layer of white or yellowish-white substance adhering to the bottom of the flask is frequently noticed. The authors have analysed both this scale and a similar substance artificially prepared, and conclude it to be an ammonium calcium meconate of the formula:

$$Ca(NH_4)C_7HO_7.2H_2O \text{ or } Ca(NH_4)C_7HO_7.3H_2O.$$

The chief importance of this salt lies in the fact that it consumes an amount of acid equivalent to one-quarter of the same weight of morphine, and hence its presence vitiates the subsequent titration of the morphine with acid. The same, or a similar salt, appears to have been noticed, but not analysed, by Robiquet in 1833 (*Ann. Chem.*, v., 82). A. G. L.

Adulterated Saccharin. R. Krzizan. (*Zeit. Untersuch. Nahr.-Genussmittel*, 1905, x., 245-247.)—Five samples of saccharin tablets examined were found to consist almost entirely of plaster of Paris. Only traces of saccharin were present, and this had evidently been dusted over the surface of the tablets. A sixth sample also consisted mainly of plaster of Paris (about 92 per cent.), the amount of saccharin present being 1·63 per cent.; the remainder consisted of calcium phosphate, calcium carbonate, and sodium hydrogen carbonate. W. P. S.

A Method of Determining Pyramidon. A. Astruc and G. Pégurier. (*Ann. de Chim. anal.*, 1905, x., 302, 303.)—The method is based upon the fact that pyramidon, like antipyrin, combines with picric acid (1 molecule : 1 molecule) to form an insoluble compound, and that the excess of picric acid can then be titrated in the way devised by Lemaire. In making an estimation 0·231 gram of the sample (molecular weight of pure pyramidon = 231) is dissolved in 10 c.c. of water, and the solution mixed with 40 c.c. of $\frac{N}{20}$ picric acid solution (11·45 grams per litre). The mixture is shaken for some minutes to promote the formation of the compound, and then filtered, and the excess of picric acid determined in 25 c.c. of the filtrate by titration with $\frac{N}{10}$ potassium hydroxide, with phenolphthalein as indicator. Four times the number of c.c. of alkali used, deducted from the original 40 c.c. (of picric acid) and multiplied by five, gives the percentage of pure pyramidon in the sample. C. A. M.

The Determination of Iron in Ferrum Redactum. A. Christensen. (*Zeit. anal. Chem.*, 1905, xliv., 535-540.)—The method of determining the metallic iron in the *ferrum redactum* of the German Pharmacopœia consists in dissolving the metal by means of iodine in potassium iodide, but the author regards this as inferior to Wilner's method (digestion with mercuric chloride), though that too is shown to be liable to give erroneous results. The method in which ferric chloride is used ($Fe + 2FeCl_3 = 3FeCl_2$) and the insoluble residue of iron oxide weighed has long been abandoned as unreliable, since part of the oxide may also pass into solution as a basic salt. Good results, however, can be obtained by titrating the resulting solution, and taking care that the ferric chloride reagent is absolutely free from acid.

It can be prepared by dissolving the anhydrous ferric chloride little by little with constant stirring in cold water. Even if a slight black insoluble residue be left, its amount can be determined once for all, and deducted in subsequent determinations. A weighed quantity (about 0·5 gram) of the *ferrum redactum* is placed in a 100-c.c. flask, from which the air has been expelled by carbon dioxide, 50 c.c. of the ferric chloride solution introduced, and the whole shaken at intervals for fifteen to twenty minutes, while the flask is either kept closed or a continuous current of carbon dioxide passed through it. The liquid is then made up to the mark with boiled water, well shaken, and allowed to stand overnight, after which 10 to 20 c.c. are withdrawn, added to 50 c.c. of 10 per cent. sulphuric acid (previously boiled and cooled in a current of carbon dioxide), and titrated with $\frac{N}{10}$ potassium permanganate solution. The tabulated results obtained by this method are closely concordant, and agree well with those calculated from the amount of hydrogen evolved on treating the substance with dilute sulphuric acid. C. A. M.

ORGANIC ANALYSIS.

Estimation of the Amount of Alcohol in Fusel Oils. R. Peters. (*Chem. Zeit. Rep.*, 1905, xxix., 218.)—The author recommends the following process as the most reliable: 100 grams of fusel oil are mixed with 50 c.c. petroleum ether, and vigorously shaken up with 100 c.c. water in a separating funnel for two minutes. The shaking with water is repeated twice, and the united clear water-alcohol extracts are distilled after addition of powdered charcoal until about 150 c.c. have passed over. The distillate is then treated with 50 c.c. petroleum ether and twice shaken for two minutes with 100 c.c. at a time of a concentrated solution of calcium chloride. Powdered charcoal is added to the calcium chloride solutions containing the alcohol, and exactly 100 c.c. is distilled off. The specific gravity of this distillate is found, and the amount of alcohol obtained from Windisch's tables. E. K. H.

The Detection and Determination of Ethyl and Methyl Alcohols in Mixtures by the Immersion Refractometer. Albert E. Leach and Hermann C. Lythgoe. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 964.)—The authors have found that the difference of the refractive indices of ethyl and methyl alcohols is so great that it may be made the basis of a method for analysing mixtures of the two alcohols. The following table gives the readings found for each per cent. of alcohol, using a Zeiss immersion refractometer (*Journ. Amer. Chem. Soc.*, 1904, xxvi., 1196), at 20° C.

In making an analysis, the specific gravity of the liquid is first determined. Since the specific gravities of methyl and ethyl alcohols and of their aqueous solutions are practically identical, the total weight of alcohol present can be calculated from this. The refractive index is next found, and the percentage of each alcohol present is then calculated by interpolation between the scale readings given in the table for aqueous solutions of each alcohol of the same strength as the sample.

The exactness of the method increases with the alcoholic strength of the sample. With 50 per cent. of alcohol, less than 1 per cent. of methyl alcohol can be readily determined, whilst with 90 per cent. of alcohol 0·1 per cent. of methyl alcohol can be determined. The presence of any volatile oils influences the accuracy of the method considerably, but mere traces do not cause any serious errors. Acetone, which has a refractive index considerably lower than that of ethyl alcohol, also exercises a marked effect; but the purified wood alcohol used as adulterant for ethyl alcohol contains so little acetone that its presence may be ignored.

Per Cent. by Weight of Alcohol.	Scale Readings.		Per Cent. by Weight of Alcohol.	Scale Readings.		Per Cent. by Weight of Alcohol.	Scale Readings.	
	Methyl Alcohol.	Ethyl Alcohol.		Methyl Alcohol.	Ethyl Alcohol.		Methyl Alcohol.	Ethyl Alcohol.
0.	14·5	14·5	34.	35·2	74·4	68.	34·0	99·4
1.	14·8	16·0	35.	35·8	75·8	69.	33·5	99·7
2.	15·4	17·6	36.	36·3	76·9	70.	33·0	100·0
3.	16·0	19·1	37.	36·8	78·0	71.	32·3	100·2
4.	16·6	20·7	38.	37·3	79·1	72.	31·7	100·4
5.	17·2	22·3	39.	37·7	80·2	73.	31·1	100·6
6.	17·8	24·1	40.	38·1	81·3	74.	30·4	100·8
7.	18·4	25·9	41.	38·4	82·3	75.	29·7	101·0
8.	19·0	27·8	42.	38·8	83·3	76.	29·0	101·0
9.	19·6	29·6	43.	39·2	84·2	77.	28·3	100·9
10.	20·2	31·4	44.	39·3	85·2	78.	27·6	100·9
11.	20·8	33·2	45.	39·4	86·2	79.	26·8	100·8
12.	21·4	35·0	46.	39·5	87·0	80.	26·0	100·7
13.	22·0	36·9	47.	39·6	87·8	81.	25·1	100·6
14.	22·6	38·7	48.	39·7	88·7	82.	24·3	100·5
15.	23·2	40·5	49.	39·8	89·5	83.	23·6	100·4
16.	23·9	42·5	50.	39·8	90·3	84.	22·8	100·3
17.	24·5	44·5	51.	39·7	91·1	85.	21·8	100·1
18.	25·2	46·5	52.	39·6	91·8	86.	20·8	99·8
19.	25·8	48·5	53.	39·6	92·4	87.	19·7	99·5
20.	26·5	50·5	54.	39·5	93·0	88.	18·6	99·2
21.	27·1	52·4	55.	39·4	93·6	89.	17·3	98·9
22.	27·8	54·3	56.	39·2	94·1	90.	16·1	98·6
23.	28·4	56·3	57.	39·0	94·7	91.	14·9	98·3
24.	29·1	58·2	58.	38·6	95·2	92.	13·7	97·8
25.	29·7	60·1	59.	38·3	95·7	93.	12·4	97·2
26.	30·3	61·9	60.	37·9	96·2	94.	11·0	96·4
27.	30·9	63·7	61.	37·5	96·7	95.	9·6	95·7
28.	31·6	65·5	62.	37·0	97·1	96.	8·2	93·9
29.	32·2	67·2	63.	36·5	97·5	97.	6·7	94·0
30.	32·8	69·0	64.	36·0	98·0	98.	5·1	93·0
31.	33·5	70·4	65.	35·5	98·3	99.	3·5	92·0
32.	34·1	71·7	66.	35·0	98·7	100.	2·0	91·0
33.	34·7	73·1	67.	34·5	99·1			

A. G. L.

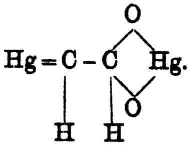
The Behaviour of Vanillin in Formaldehyde Tests. C. H. La Wall. (*Amer. Journ. Pharm.*, 1905, lxxvii., 392-394.)—A sample of ice-cream, reported to contain formaldehyde, was examined by the author, and found to give unmistakable reactions with Hehner's test, the resorcinol-sulphuric acid test, and the phenol-sulphuric acid test, but negative results with the phenylhydrazine test, and the phloroglucinol, resorcinol-soda, and hydrochloric acid tests. Vanillin was recognised as present by its flavour, and comparative tests were therefore made with a solution of vanillin (1 : 1,000), which it was found produced zone reactions closely similar to those produced by formaldehyde. Coumarin gave negative results in all the tests. The following table shows the comparative results obtained :

	Milk.	Milk and Formaldehyde, $\frac{1}{20000}$.	Formaldehyde, $\frac{1}{20000}$.	Coumarin, $\frac{1}{1000}$.	Vanillin, $\frac{1}{1000}$.	Milk with Vanillin, $\frac{1}{10000}$.
Phenol test.	No reaction.	Pink zone.	Pink zone.	No reaction.	Pink zone.	Pink zone.
Resorcinol test.	No reaction.	Pink zone.	Pink zone.	No reaction.	Pink zone.	Pink zone.
Hehner's test.	No reaction.	Violet zone.	Violet zone.	—	Violet zone.	Violet zone.
Phloroglucinol test.	—	—	Cherry-red colour.	No reaction.	No reaction.	—
Phenylhydrazine test:						
(*a*) With sodium nitro-prusside.	Yellow.	Green.	Blue.	No reaction.	No reaction.	Yellow.
(*b*) With potassium ferrocyanide.	—	—	Red.	No reaction.	Pale red	—
Hydrochloric acid test.	Brownish-rose.	Rose-purple.	—	—	—	Brownish-rose.
Resorcinol-soda test.	Yellowish-brown.	Red.	Red.	No reaction.	No reaction.	Yellowish-brown.

C. A. M.

The Action of Aldehydes upon Mercuric Oxide in the Presence of an Alkali. A. Leys.

(*Journ. Pharm. Chim.*, 1905, xxii., 107-112.)—Ordinary aldehyde has no action upon mercuric oxide in aqueous solution, even after the addition of sodium or potassium hydroxide, but on introducing a small amount of a neutral salt that has a solvent action upon the mercuric oxide—*e.g.*, sodium chloride or sulphite—a dense white precipitate is obtained. This reaction takes place with all aldehydes containing the group $CH_2 - COH$, whereas formaldehyde, furfural, aldoses, and certain aromatic aldehydes which do not contain this group cause only a fine deposit of metallic mercury. The white compound formed is insoluble in water and alcohol, and in hot dilute sulphuric acid, but dissolves readily in a solution of potassium cyanide and in dilute hydrochloric acid, and with difficulty in hot dilute nitric acid. Its formula, according to the results of elementary analysis, is :



A reagent for differentiating aldehydes by this reaction is prepared by gently heating 1 gram of powdered mercuric oxide with 100 c.c. of a 5 per cent. solution of sodium sulphite until solution is complete, and filtering the liquid when cold, with precautions to avoid contact with any ammonia vapour. The filtered liquid can be kept for a long time unaltered. In making a test 10 c.c. of the reagent are mixed with a few drops of the aldehyde solution, and then with an equal volume of dilute potassium hydroxide solution (laboratory solution with nine times its volume of water), and the mixture allowed to stand for a short time. If necessary, a small proportion of alcohol may also be added to bring insoluble aldehydes into solution. The reaction might be used to detect aldehydes in the alcoholic extracts of certain plants, provided precautions be taken to eliminate terpenes, which also yield insoluble compounds with the mercuric reagent. Acetone gives no reaction in the cold, but a white precipitate is produced on warming the liquid. A dense white precipitate is given by acetylacetone, even in the cold, after shaking the mixture for a short time. Acids with an ethylene grouping, such as oleic and crotonic acids, give no reaction, while other acids, such as formic acid, or containing a secondary alcohol group, such as tartronic acid, give a deposit of mercury. C. A. M.

Determination of Metacresol in Cresol Disinfectants. Wesenberg. (*Pharm. Zeits.*, 1905, l., 280; through *Pharm. Journ.* 1905, lxxv., 229.)—As metacresol has a markedly higher antiseptic value than either ortho- or para-cresol, its determination in a mixture of cresols in soaps or disinfectants is a matter of importance. For this purpose Rushig's method may be employed, the procedure being as follows: 10 grams of the substance are treated in a litre flask with 17 c.c. of concentrated sulphuric acid, and kept at a temperature of 100° C for one hour; 90 c.c. of nitric acid (specific gravity 1·30) are then added in one portion, and the heating cautiously continued on a water-bath. When nitrous fumes cease to be evolved, ortho- and para-cresol will have been entirely destroyed. Forty c.c. of water are now added, and the contents of the flask allowed to cool for two hours. The crystalline cake, which will contain fatty acids as well as trinitro-cresol if soap were present in the original substance, is then removed and extracted with light petroleum. The fatty acids are dissolved, whilst the trinitro-cresol remains quite insoluble, and may be collected, dried, and weighed. Each 1·74 gram of trinitro-cresol corresponds with 1 gram of metacresol. W. P. S.

The Testing of Oil of Turpentine. E. Valenta. (*Chem. Zeit.*, 1905, xxix., 807.)—In the dry distillation of colophony for the purpose of obtaining "resin oil" and "fused resin"—products which are used in the production of printing inks—there is obtained, as a by-product, "essence of resin" (pinolin), this name being given to the substances distilling over before the proper resin oil. The pinolin so obtained is a brownish to dark-brown liquid of peculiar smell, somewhat resembling wood-tar, and having a strong acid reaction. The crude pinolin contains impurities, acids and creosote-like bodies, in greater or less amount, from which it can be freed by successive treatment with caustic soda, water, sulphuric acid, very dilute caustic soda, and repeated washing. After this treatment and redistillation, pinolin is a mobile, clear, colourless or slightly yellow liquid of a not unpleasant smell, which begins to boil about 70° C., and the chief part of which distills over between 160° and 220° C. This was at one time used as an illuminant, but at the present time there are obtained from it "essence of turpentine" (which serves as a substitute for oil of turpentine, or is sold mixed with oil of turpentine in place of the pure oil), and "light resin oil," which is used as a solvent for resin in the manufacture of varnish, etc.

The author has investigated various pinolins obtained from different sources in order to determine the behaviour of those fractions which seem suitable for the adulteration of turpentine. For the detailed figures, reference must be made to the original. The main conclusions are as follows: The fractions boiling below 170° showed a negative rotation, increasing slightly with rise of boiling-point, whilst those boiling above this temperature have a positive rotation, also increasing with rise of boiling-point; the refractive indices and specific rotations of the lower boiling fractions, which serve as "essence of turpentine" for the adulteration of true oil of turpentine, are so nearly the same as those of true oil of turpentine that these constants cannot serve to detect their addition.

These fractions, however, show Herzfeld's sulphurous acid reaction (yellow coloration on shaking with a solution of sulphurous acid), which is not given by true oil of turpentine or light petroleum.

Herzfeld's reaction alone is insufficient to demonstrate the addition of "essence of turpentine" or pinolin, as it is given also by resin, "resin oil," and "pine-resin oil." By fractionating the oil, and using for Herzfeld's test only the portions boiling below 170° C., the first two are eliminated, and a yellow colour indicates essence of turpentine or pine-resin oil, but does not distinguish between these.

The author, therefore, extended his observations, and gives the following reactions as important for the "essence of turpentine": (1) Acetic anhydride and one drop of sulphuric acid give an intense green colour; (2) 1 part of oil with 1 to 2 parts of a 6 per cent. solution of iodine in chloroform or carbon tetrachloride, warmed gently on a water-bath, give intense green to olive-green colours. The strongest colours are given by the lower boiling fractions. This last reaction is not given by any of the following: Oil of turpentine, pine-resin oils, light petroleum, oil of camphor, resin oil.

Under "pine-resin oils" are included less valuable turpentines, which are obtained from roots rich in oil and resin, from pine-wood and similar materials, and rectified. The oils of this group, to which also the more or less yellow coloured commercial turpentines sold under the name of "Polish," "Russian," or "Hungarian" turpentine belong, are distinguished by a peculiar sharp smell which is not lost by treatment with lime or alkalies and repeated distillation. They have a stronger action on the skin than true oil of turpentine; they contain, at least if not rectified, much free acid; they dissolve asphalt more readily than pure rectified oil of turpentine.

The author gives a table showing the results obtained by the iodine solution test, by the addition of 1 part of a 1 per cent. gold chloride solution to 1 part of oil, and giving the value of the refractive index and specific rotation for true oil of turpentine from different sources, for various pine-resin oils, and for pinolin, oil of camphor, resin oil and purified petroleum. For details reference must be made to the original. The most noteworthy facts are (1) the olive-green coloration with the chloroform solution of iodine is given by pinolin alone; (2) only pure oils of turpentine give no decolorization in the water-layer of gold chloride.

The specific rotation is very variable and of no value as an indication of purity or the reverse; the refractive index is in general lower for true oil of turpentine, but the differences are not great enough to be of much use. E. K. H.

The Conversion of Dextrose into Lævulose, and the Determination of Lævulose. H. Ost. (*Zeit. angew. Chem.*, 1905, 1170.) When dextrose is digested with cold sulphuric acid, the sugar is converted into lævulose in appreciable proportions.

The author dissolved 1·1 kilograms (= 1 kilogram anhydrous) of crystallized dextrose in a cold mixture of 2 litres of water and 1 litre of sulphuric acid, and allowed the liquid to stand for four months at room temperature. The brown fluid was then neutralized with pure calcium carbonate, filtered, and evaporated at a moderate temperature. A thick syrup was obtained, from which a large portion of the unaltered dextrose crystallized out. The separation of crystals from the mother liquor was pushed as far as possible by means of fractional solution in absolute alcohol, combined with crystallization. Finally, a mother liquor rich in lævulose was obtained, with a rotatory power of $[a]_D - 10$, and the author calculates that this represents 5 to 10 per cent. of lævulose.

The determination of lævulose in a mixture of unknown composition is a very difficult matter, as it never crystallizes out from an impure syrup, it yields no hydrazone that can be isolated, and its osazone is identical with that obtained from dextrose and mannose.

The best method of determining lævulose is that of Dubrunfaut, depending on the formation of a calcium compound, but the results should only be accepted with caution, and confirmed, if possible, by a different method.

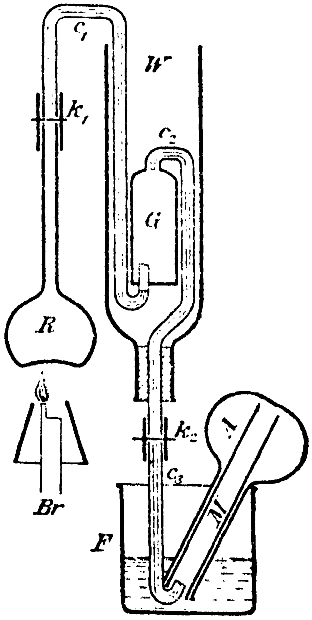
A 10 per cent. solution of the sugar is cooled in a freezing mixture till it is partly frozen, and six parts of dry sifted calcium hydrate to ten parts of the sugar are added, with careful shaking. If a large proportion of lævulose is present, a stiff mass of fine needle crystals forms in a few moments. After a quarter of an hour, the small portion of liquid is poured off, and the mass of crystals washed with ice-water, and decomposed by oxalic acid, which is cautiously added with small pieces of ice till there is a faint acid reaction after repeated shaking. The filtrate is evaporated at 50° to 60° C. In this way, 20 grams of a commercial invert sugar, $[a]_D - 15°$, and containing 4 per cent. cane-sugar, yielded 8 grams of crude lævulose of $[a]_D - 73°$, or 40 per cent. In doubtful cases, the result should be checked by Sieben's method (*Chem. Zuckerarten*, 1904, 895), and by a preparation of the osazone.

The author discusses at some length the preparation and properties of this latter, but is of opinion that it is of very limited use in determining the presence of small percentages of lævulose. If any crystals of the osazone appear in the cold within twenty hours, lævulose is certainly present, but a negative result is inconclusive.

The methyl phenylosazone obtained from dextrose and lævulose is identical in every respect. It crystallizes from hot benzene in long orange-coloured needles of fairly sharp melting-point (155° to 158°) and is quite insoluble in water. It has the specific rotatory power $[\alpha]_D + 48$. A. N. C.

Determination of Nitric Acid in the Presence of Organic Substances.

B. Pfyl. (*Zeit. Untersuch. Nahr.-Genussmittel,* 1905, x., 101-104.)—The method is based on the reduction of the nitric acid to nitric oxide by means of ferrous chloride and hydrochloric acid; the nitric oxide is washed by passing it through sodium hydroxide solution and absorbed by potassium permanganate solution. The reduction is carried out in a flask of about 75 c.c. capacity, *R* (see illustration), which has a long, narrow neck. The latter is connected by a piece of indiarubber pressure tubing and a clamp, k_1, to the delivery tube, c_1. The solution containing the nitric acid (nitrate) is placed in the flask and boiled down to a volume of about 5 c.c., the end of the delivery-tube being placed under water. When the flame is removed, the water fills the tube and begins to enter the flask. At this moment the clamp, k_1, is closed, and the contents of the flask allowed to cool somewhat; 20 c.c. of a solution containing 30 grams of ferrous chloride in 50 c.c. of dilute hydrochloric acid (2 : 3) are allowed to enter the flask, which is then heated, after closing the clamp, in a boiling water-bath for about forty-five minutes; 10 c.c. of boiled dilute sulphuric acid (1 : 4) are then introduced, the end of the delivery-tube is closed with a Bunsen valve and placed under the bell-jar, *G*. The latter has a capillary tube, c_2, leading from the top, as shown. The bell-jar and the outer vessel, *W*, are filled with boiled 15 per cent. sodium hydroxide solution. By opening the clamp, k_2, the level of the soda is adjusted until the solution just covers the bell-jar. The tube, c_3, dips below the neck of the flask, *A*, which is filled with boiled sulphuric acid (1 : 4) and inverted in the vessel, *F*, containing dilute sulphuric acid. The clamp, k_1, is now opened and the contents of the flask, *A*, cautiously warmed. The evolved gas is collected in the bell-jar, and from its volume the amount of $\frac{N}{10}$ potassium permanganate to be added to the vessel *F* is judged. On opening the clamp, k_2, the gas bubbles into the flask, *A*. The contents of the flask, *R*, are then boiled until no more gas is evolved. The flame is then removed and the vessel, *W*, filled up with sodium hydroxide solution, when all the gas in the bell-jar is driven over into the flask, *A*. The tube, c_3, is now taken out and rinsed with water. By carefully shaking the flask, *A*, keeping the neck below the surface, the mixture of the sulphuric acid and permanganate is attained,



and the whole of the gas is absorbed. The tube, *M*, aids the outflow of the acid displaced by the gas. After a time the excess of $\frac{N}{10}$ permanganate solution is titrated back with $\frac{N}{10}$ ferrous chloride solution. $\frac{N}{10}KMnO_4$ corresponds with $\frac{1}{30}KNO_3$ or $\frac{1}{60}N_2O_5$. The apparatus is made by Bender and Hobein, Munich. W. P. S.

Estimation of Glycuronic Acid. B. Tollens. (*Chem. Zeit. Rep.*, 1905, xxix., 219.)—The author disputes the statements of C. Neuberg and W. Neimann that combined glycuronic acids change on distillation with hydrochloric acid, and therefore do not give the theoretical quantities of furfurol. He calls attention to the fact that the method, if by no means perfect, yields approximate figures, and is also applicable to euxanthic and urochloralic acids; in the case of the latter the saccharic acid method failed to give a result, whilst from euxanthic acid Neuberg and Neimann obtained only 60 to 70 per cent. of the calculated quantity of saccharic acid. According to Neuberg and Neimann the saccharic acid method is really useful only for phenylglycuronic acid. Consequently, as soon as the pure combined glycuronic acids are separated, the furfurol distillation method is serviceable for the approximate estimation of the glycuronic acid occurring therein, and is probably more useful than the saccharic acid method, the application of which seems limited. For the determination of glycuronic acid in urine, or in mixtures which contain pentosanes, pentoses or nucleoproteids, the author's method is not applicable, and he has never recommended it for these cases. E. K. H.

Determination of Sulphur and Phosphoric Acid in Foods, Fæces, and Urine. W. L. Dubois. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 729-732.)—Since the publication of a previous communication (ANALYST, 1905, 90) some important changes have been made in the methods employed by the Bureau of Chemistry (U.S.A.) for the determination of sulphur and phosphoric acid in foods, etc. Urines are still burnt according to Osborne's method, but for foods and fæces the following modification of Neumann and Meinertz's process is used: Four grams of fresh foods, 2 grams of dried foods, or 1·5 grams of dried fæces, are placed in a 100-c.c. nickel crucible. The dry charges are moistened with 2 c.c. of water, 5 grams of anhydrous sodium carbonate and 5 grams of sodium peroxide are added, and the whole well mixed. With fæces, the sodium peroxide may be added all at once, but with dried foods one-half is stirred in; and then, after standing a time, the remainder is added. The crucible is placed over a low alcohol flame until the material is dry; the flame is then increased, and the charge heated until it disintegrates and can be easily broken into a powder. A further quantity of sodium peroxide is now added, and the mixture fused, increasing the flame at the end to a fierce blast. When the liquid state is reached, a little more peroxide may be added to insure complete oxidation. The fused mass, after cooling, is dissolved out and treated exactly as described for the Osborne method. W. P. S.

A Rapid Method of determining Chlorine in Urine. W. M. Dehn. (*Zeit. physiol. Chem.*, 1905, xliv., 11; *Chem. Zeit.*, 1905, xxix. [*Rep.*], 173, 174.)—The following modification of Volhard's method is stated to give more accurate results than the methods ordinarily employed: 10 c.c. of the urine are mixed with a few grams of sodium peroxide and evaporated on the water-bath. The residue is treated with 10 c.c. of water and sufficient dilute nitric acid to render the liquid just acid to litmus-paper, after which the chlorine is titrated by Volhard's method, a little iron nitrate being previously introduced as indicator. The best method of titrating is to have equivalent solutions of potassium thiocyanate and silver nitrate, the latter containing 29·075 grams per litre (1 c.c. = 0·01 gram of sodium chloride). A few drops of the thiocyanate solution are first run in from a burette, until the liquid has an intense red colour. If this disappears it shows that the nitric acid or iron nitrate contained nitrous acid, or that the evaporation on the water-bath was incomplete, so that some hydrogen peroxide was left; this would cause an error in the titration. After the addition of the thiocyanate the liquid is thoroughly stirred and then slowly titrated until the red colour disappears. The difference between the amount of thiocyanate and silver nitrate solutions used gives the amount of sodium chloride in grams per litre. C. A. M.

A Further Report on Parr's Process for the Determination of the Heat of Combustion. G. Lunge and H. Grossmann. (*Zeit. angew. Chem.*, 1905, 1249.)—This method for the determination of the heat of combustion in coals depends on oxidation in a closed vessel with sodium peroxide, and the comparative simplicity of both the process and apparatus gives it a great advantage over the ordinary oxygen bomb method. Unsatisfactory results have, however, been obtained by more than one operator, and there is one obvious source of error to be feared—that the reaction will be either explosive or incomplete.

By a few slight modifications this difficulty can be generally avoided, and results obtained that agree closely with those yielded by the best bomb methods. There remains the disadvantage that an empirical coefficient has to be used for the calculation of the result, and this must be determined for each fresh apparatus. In the modified process, a mixture of exactly 0·5 grams coal, 0·5 grams tartaric acid, and 10 grams sodium peroxide is employed.

The coal and the sodium peroxide must be finely powdered and intimately mixed. The peroxide should be of the best possible quality, and previously tested by the combustion of 0·5 grams tartaric acid. This should cause a rise of temperature of almost exactly 0·832°. The figure obtained must be subtracted from that observed in the calorimeter, and this will include the correction for the hot wire by which the combustion is started.

A correction for the hygroscopic water is, for coals, generally superfluous.

The rise in temperature remaining over for the combustion of the coal is multiplied by the coefficient 1,540 per 1 gram of coal, and the product taken as the heat of combustion of the coal.

This figure 1,540 is, of course, only of use with a Parr's calorimeter of the standard type, with a water content of 2,000 grams and a water value of 2,123. With other values the corresponding coefficient must be found by calculation.

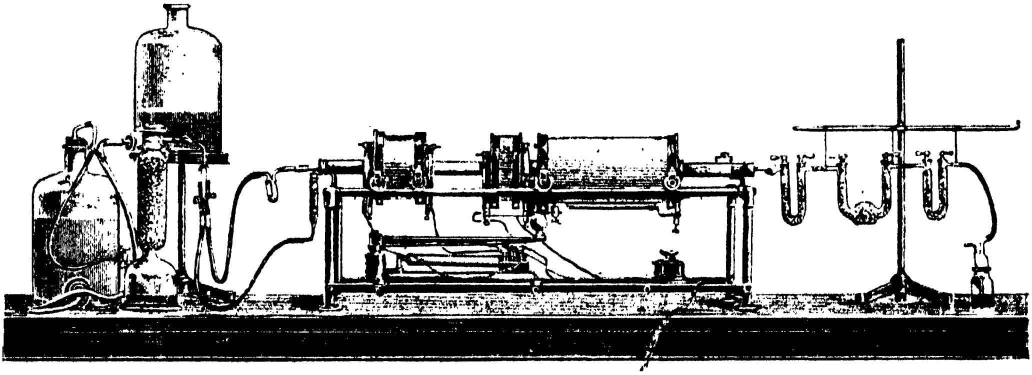
Finally, the method is not recommended for coals with a calorific value of less than 7,500, particularly lignites. A. N. C.

Simplified Ultimate Analysis, and its Employment in Technical Work. M. Dennstedt. (*Zeit. angew. Chem.*, 1905, 1134.)—The author has previously published (*Zeit. angew. Chem.*, 1904, 30) the principle and details of his method—combustion in a double current of oxygen, without any admixture with copper oxide, and only describes here a modified apparatus by which his process is considerably shortened.

A special form of the Heraus electric furnace has been designed, and is so built that one part serves for the heating of the rolled platinum foil that is used as the contact substance, while only so much current is used elsewhere as is required to heat the combustion-tube to 300° C.

A nickel trough carries sufficient heat from the furnace to keep the exposed parts of the tube hot enough to prevent any condensation of water.

The illustration shows the apparatus. From left to right are the oxygen vessels, the drying tower, beyond which the current of gas separates, one part passing through a small bulb filled with sulphuric acid into the capillary of the tube leading over the substance to be analysed, and the other through a short calcium chloride tube directly into the combustion-tube. On the right is the ordinary absorption apparatus.



With this apparatus almost every kind of substance can be dealt with.

Unstable organic compounds should be mixed with an inert substance such as glass powder, and mineral oils with pure ignited kieselguhr, to avoid any explosive action. By forming an air-bath round that part of the tube containing the sample the author has even succeeded in dealing with gun-cotton, its temperature not rising above 160° C., and the combustion being prolonged over twelve hours.

The method also provides an accurate and rapid means for the estimation of sulphur in pyrites or other material.

The sulphur dioxide is absorbed with calcined soda in a small boat in the tube, unless the carbon and hydrogen are being determined at the same time, when lead superoxide is used.

To insure complete absorption, the whole length of the combustion-tube must be kept at 300° C. For this purpose a special burner with twenty small blue flames on a horizontal tube is used. The estimation of sulphur in pyrites by this method yields results rather higher than those obtained by the generally-used Lunge's method, but the author gives reasons for believing that the higher figures are the more accurate. A. N. C.

INORGANIC ANALYSIS.

Quantitative Separations in Presence of Hydroxylamine. P. Jannasch and W. Cohen. (*Chem. Zeit. Rep.*, 1905, xxix., 248.)—The authors have proved that the action of hydroxylamine depends partly on its great reducing power and partly on the formation of characteristic double salts, as well as on the increase of the insolubility of the ammonia precipitate in excess of the precipitant. Usually hydrochloric acid solutions are used: presence of sulphuric acid necessitates more prolonged washing, but nitric acid is completely indifferent. The presence of formic acid does not hinder the precipitation, but acetic acid can only be used as solvent with certain precautions. Oxalic, citric, and tartaric acids hinder the precipitation either completely or in part. The separation of aluminium and chromium from copper, and that of iron from copper and zinc, are described. E. K. H.

Separation of Iron from Manganese and Magnesium, and of Aluminium and Chromium from Manganese, Zinc, Nickel, and Magnesium by Hydroxylamine in Ammoniacal Solutions. P. Jannasch and F. Rühl. (*Chem. Zeit. Rep.*, 1905, xxix., 248.)—The property of manganese of not being precipitated by ammonia in the presence of hydroxylamine even on continued boiling has led to a method of separating iron from manganese. For this purpose about 0·3 to 0·4 grams of Mohr's salt (ferrous ammonium sulphate) and about the same weight of manganese ammonium sulphate were dissolved, together with 5 to 6 grams of hydroxylamine hydrochloride, in hot water and 5 to 6 c.c. concentrated hydrochloric acid. Thirty c.c. of concentrated ammonia were added to the hot colourless solution: a fine powdery bright-red precipitate was obtained, for the filtration of which a double filter was required. The precipitate was washed, dried, ignited and weighed as Fe_2O_3. The filtrate was evaporated to dryness, the residue gently heated to drive off salts of ammonium and hydroxylamine, and the manganese then estimated by the hydrogen peroxide method due to one of the authors. The separation of iron from magnesium was exactly similar, except that a second precipitation was advisable, as one precipitation did not completely separate the metals.

A similar procedure was used for the separation of aluminium and chromium from manganese, zinc, nickel, and magnesium. Details of the experiments may be found in the original paper (*Journ. prakt. Chem.*, 1905, lxxii., 1). E. K. H.

Quantitative Separation of Iron and Thorium from Uranium. P. Jannasch and J. Schilling. (*Chem. Zeit.*, 1905, xxix., 248.)—For the separation of thorium from uranium, a quantity of the salts of the two metals (the pure nitrates) was dissolved in hot water with five times the amount of hydroxylamine hydrochloride, and concentrated ammonia was then added to the boiling solution. A brisk evolution of nitrogen took place, and a white voluminous precipitate of hydrated thoria came down. The thoria was weighed as oxide in a platinum crucible. The filtrate was evaporated to dryness, and heated to volatilize the hydroxylamine hydrochloride; the residue was taken up with dilute hydrochloric acid, and the uranium precipitated by ammonia at the boiling-point. The uranium precipitate was converted to UO_2 by heating in a current of hydrogen and weighed as such. E. K. H.

The Separation of Iron and Aluminium from Manganese, Calcium, and Magnesium in the Analysis of Plant Ashes. P. Kaschinsky. (*Chem. Zeit. Rep.*, 1905, xxix., 217.)—As plant ashes usually contain much phosphoric acid and less iron and aluminium than is necessary for combining with the phosphoric acid, it is necessary to add sufficient ferric chloride to transform all the phosphoric acid to iron phosphate before precipitating with ammonia. This method gives accurate results, but is somewhat complicated; the simpler processes due to König and to Tollens have, therefore, been often used in preference. The author has now tested whether these two latter methods give correct figures, and has found that in both processes calcium phosphate may be carried down with the iron phosphate, and in considerable quantity. The amount of calcium phosphate carried down depends upon the conditions of the analysis and on the composition of the ash analysed—*e.g.*, on the quantity of free acetic acid, on the amounts of calcium and of phosphoric acid, and on their relative proportion. Accurate results were obtained only by using the old method of adding ferric chloride. E. K. H.

The Titration of Ferrous Iron with Permanganate in the Presence of Hydrochloric Acid. Gregory Paul Baxter and Harry Louis Frevert. (*Amer. Chem. Journ.*, 1905, xxxiv., 109.)—From a study of the titration of ferrous iron by potassium permanganate in the presence of hydrochloric acid (without the addition of sulphuric acid), the authors conclude that, even at the highest possible temperatures, it is impossible to obtain accurate results unless manganous salts are present, probably owing to the production and volatilization of hypochlorous acid. If the titration is carried out at 80° to 90° C., however, the results show a nearly constant positive error of only about 0·3 per cent. Manganous sulphate is more effective than the chloride, and at low temperatures more of the salt is required than at high temperatures. A. G. L.

Electrolytic Method of Estimating Zinc. K. Jene. (*Chem. Zeit.*, 1905, xxix., 803.)—The method is briefly as follows: The zinc must be present as sulphate. To a neutral or slightly acid solution 3-4 grams of caustic soda are added, and the solution electrolysed at 50° C. by a current of 1 ampère. The E.M.F. required for ordinary proportions (using a Classen's basin and 120-140 c.c. of liquid) is then 3·8 to 4·3 volts. The time necessary for the electrolysis is about two hours for solutions containing 0·2 to 0·25 gram zinc. The advantage of this process over other electrolytic methods for estimating zinc lies in the fact that the previous separation of any accompanying metals (Fe, Al, Mn) is not necessary, whilst the results are equally accurate. The author performed test experiments with known quantities of zinc in solutions containing iron, aluminium, and manganese sulphates in varying quantities, and found in all cases the exact theoretical amount of zinc. The precipitated zinc was also tested and found to be perfectly pure. The hydroxides precipitated by the caustic soda float in the basin during the electrolysis without causing any disturbance: in the presence of much iron the electrolysis took somewhat longer, but in no case was more than two hours twenty minutes necessary for complete separation of the zinc.

For the analysis of the products mentioned above, the author proceeded as follows: 0·5 gram of the substance was dissolved in aqua regia, evaporated to dryness, and again evaporated with 1-2 c.c. of dilute sulphuric acid (1 : 1). After cooling, the residue was diluted with water, boiled, filtered, and the precipitate washed with hot water—with skilful working the total filtrate is never more than 80 to 100 c.c., and is immediately suitable for electrolysis—4 to 7 grams of caustic soda were added to the liquid, and allowed to dissolve completely. The solution of sodium zincate, after warming to about 50° C., became clear, the hydroxides floating about in the liquid, and was poured into the electrolysing basin, which was a copper-plated platinum basin. The temperature was maintained approximately at 50° C. during the electrolysis: the current was 1 ampère; the E.M.F. 3·8 to 4·2 volts. The whole of the zinc was precipitated in from one and a half to two hours. The end of the action was determined by hanging over the edge of the dish a clean strip of copper, and observing whether any deposit of zinc occurred thereon after fifteen minutes. The basin was washed out in the well-known manner without interrupting the current; it was then well washed with cold water, and then with absolute alcohol free from acid, and dried in the air-bath for one to two minutes, allowed to cool in the desiccator, and weighed. The precipitate was in all cases blue-white in colour and coherent. In a few instances the edge was somewhat dark coloured through slight oxidation, due to evaporation of the liquid during electrolysis. To avoid this, a few drops of water should be allowed to run down the edge of the basin from time to time. The deposit of zinc can be removed by very dilute nitric acid without damaging the copper plating. The time occupied by the whole process is about five hours. The author gives some figures obtained in analyses of calamine, in which the results of the electrolytic process are compared with those of a gravimetric analysis. The figures show very fair agreement. The author considers his experience shows that the method is quite accurate enough for all ordinary technical purposes, but is not prepared to say that it is reliable for use in laboratories in zinc works. The chief merit lies in the short time occupied, and this can be further curtailed by using several platinum dishes, or even nickel dishes. E. K. H.

Zinc Dust. William Ramsay. (*Chem. News*, 1905, xcii., 80.)—Zinc dust is generally packed in old petroleum casks, from which it abstracts a relatively large amount of petroleum of a particularly disgusting odour, which is evolved when the dust is heated. The evolution of ammonia on heating zinc dust with caustic soda is probably due either to a reduction of nitrates present in the soda, or to the zinc dust having absorbed ammonia from the laboratory air, or to a combination of both causes, and not, as has been suggested, to a fixation of atmospheric nitrogen by the zinc dust. Zinc dust is sometimes pyrophoric. Under certain conditions it may be fused to a curious crystalline mass, easily powdered, and very brittle. If the oxide present be first removed by acid and washing, the dust can be smelted to metal if air be excluded. A. G. L.

A New Sensitive Reagent for Nickel. L. Techugaeff. (*Chem. Zeit. Rep.*, 1905, xxix., 247.)—The author has found an extraordinarily delicate and characteristic reagent for nickel in α-dimethylglycoxime. The solution to be tested is first freed from excess of mineral acid by the addition of ammonia or sodium acetate; some of the powdered reagent is sprinkled in, and the solution heated for a short time at the boiling-point. If the solution is not too dilute, there is formed immediately a beautiful scarlet precipitate of the composition $NiD.DH_2$ (where D = dimethylglycoxime). The reaction is so delicate that it is given very plainly by a solution of 1 part nickel to 400,000 parts water. E. K. H.

The Recognition of Sodium and Quantitative Estimation of its Compounds in Presence of those of Lithium by Means of Hydrosilicofluoric Acid. C. Reichard. (*Chem. Zeit.*, 1905, xxix., 861.)—The alkali metals are distinguished generally from others by the relatively small number of characteristic reactions they possess; and among the alkalis, sodium and lithium are those which afford specially few distinguishing properties. Any fresh reaction of these elements should be welcome, but especially one which can be used to distinguish the two closely related elements, sodium and lithium. Such a method is, in the author's opinion, provided by the use of hydrosilicofluoric acid. The author, in the course of his investigations on the silicofluorides of the alkalis, was struck by the slight solubility of the sodium salt, and was led to examine the action of hydrosilicofluoric acid on lithium compounds. As a result, it was found that no turbidity was produced on adding this to salts of lithium even in concentrated solution, and after standing for twenty-four hours still no precipitate formed. Sodium is precipitated as silicofluoride in solutions containing as little as 0·25 per cent. of its salts. These facts indicate that the method of precipitation by hydrosilicofluoric acid is a very promising one where the solution contains sodium and lithium compounds only; but it is not applicable, without modification, to the more common case where potassium is also present, as the latter is also precipitated as silicofluoride. The presence of potassium, in minerals at least, often implies the presence of one or both of the metals rubidium and cæsium—a fact which cannot be left out of count. The author proceeds to discuss the common and important case where sodium, lithium, and potassium are present. For this he recommends the following process as giving good results in the hands of an experienced and skilful worker.

The potassium and sodium are precipitated from as concentrated a solution as possible, in the form of silicofluorides, by hydrosilicofluoric acid, and the precipitate so obtained is filtered off, washed, and dried. The chief part of the powdery residue is transferred to a weighed platinum dish, the filter ignited, and the ash added to the rest of the precipitate in the dish, and the whole weighed. The mixed silicofluorides are then transformed to sulphates in the usual manner by means of concentrated sulphuric acid, and the mixed sulphates weighed. These are then dissolved in boiling water, and the total sulphate estimated by means of barium chloride. The amounts of potassium and sodium can now be calculated from the weights of mixed sulphates and of the total sulphate found by the ordinary methods of calculation for indirect analyses.

This calculation does not use the weight of the mixed silicofluorides, and this is not, therefore, absolutely necessary. But from the weights of the silicofluorides and of the sulphates one can, by a similar calculation, find the quantities of sodium and potassium without finding the amount of sulphate with barium chloride. By using the two methods a check is afforded.

The author then comments on alternative methods. If the metals are present as sulphates, the silicofluoride process of separation and estimation is specially to be recommended; an indirect estimation by precipitation of the sulphuric acid cannot be made, as the barium sulphate always carries down lithium with it in considerable amount.

The platinum chloride method would separate off cæsium and rubidium as well as platinum, but, according to Jenzsch, the method is inapplicable in the presence of lithium, as there is obtained a lithium containing precipitate, probably a mixture of K_2PtCl_6 and $KLiPtCl_6$. Whether this difficulty can be obviated can only be settled by further exact and exhaustive investigation. In any case, the method cannot meanwhile be used.

The picric acid method, on account of the relatively great insolubility of potassium picrate, can be used with advantage to separate potassium (rubidium and cæsium) from lithium, but as one must use, in order to get a sufficiently concentrated solution, the sodium salt instead of picric acid itself, the method is inapplicable where sodium is also present. It might still be used, however, if a weighed quantity of pure sodium picrate were employed.

The only other method is that of precipitation of potassium by addition of tartaric acid. This gives better promise, as tartaric acid has a solubility sufficient to enable it to be used directly as a precipitant of potassium. The only point that requires investigation is what influence the excess of tartaric acid exerts on the subsequent separation of sodium from lithium by hydrosilicofluoric acid—*i.e.*, the effect on the solubility of sodium silicofluoride. The author proposes to discuss this question in a further communication, as well as the more exact conditions for the separation of sodium and lithium. E. K. H.

The Determination of Sulphites by Iodine. R. Harmon Ashley. (*Zeits. anorg. Chem.*, 1905, xlvi., 211.)—Continuing his work on the subject (ANALYST, 1905, xxx., 285), the author finds that fairly good results may be obtained in the titration of sulphites by iodine if Rupp's method (*Ber.*, xxxvii., 409) is modified as follows: To the sulphite solution containing 1 gram of sodium bicarbonate at least twice the theoretical quantity of standard iodine solution is added, the total volume of the solution being kept below 100 c.c. The liquid is next made slightly acid with hydrochloric acid, and the excess of iodine titrated with thiosulphate. The necessity of using a fairly large excess of iodine seems to be due to the formation of dithionate if the concentration of the iodine is too small. With quantities of sulphur dioxide equivalent to from 0·1 to 0·3 gram of iodine, a number of test-analyses by this method show errors of about 1 per cent. A. G. L.

The Detection of White Phosphorus in Commercial Phosphorus Sulphide. L. Vignon. (*Bull. Soc. Chim.*, 1905, xxxiii., 805-815.)—Phosphorus sulphide, P_4S_6, has been used for many years in place of white phosphorus in the French national match factories, with the result that the "necrosis" produced by phosphorus poisoning in the workpeople has disappeared. It is essential, however, that the phosphorus sulphide should be free from white phosphorus, and the author has therefore examined the various methods of detecting it. The ordinary qualitative methods are useless for the purpose, and the appearance of phosphorescence cannot be regarded as an infallible sign, since zinc sulphide and many other substances free from phosphorus also phosphoresce. Mitscherlich's method is not applicable in the case of phosphorus sulphide, owing to the fact that traces of the pure sulphide are decomposed, with the liberation of free phosphorus (or a substance resembling it) during the distillation with water. Methods based on differences in solubility in different solvents (glycerin, benzene, turpentine oil) are also ineffectual.

Useful indications may be obtained by a determination of the boiling-point under reduced pressure. Thus, in the author's experiments, under a pressure of 30 millimetres, pure phosphorus sesquisulphide distilled at 250° to 254° C., and the commercial sulphide at the same temperature, whilst a mixture of pure sulphide with 26·4 per cent. of white phosphorus began to distil at 160° C., the temperature slowly rising to 260° C. In a mixture containing 1·8 per cent. of white phosphorus the temperature rose to 250° C. Blondlot's method of expelling the phosphorus in a current of hydrogen cannot be used, since the pure sulphide undergoes some change in the hydrogen apparatus and behaves like phosphorus. Dalmon's modification of this method is the only one that has been found applicable. This consists in passing the hydrogen over the phosphorus sulphide, and is capable of detecting the presence of at least 2 per cent. of white phosphorus. The hydrogen is phosphorescent in the dark, and burns with a green flame yielding phosphoric acid, which can be condensed and identified. The pure sulphide and the industrial sulphide should give absolutely negative results. C. A. M.

Analysis of a Bueb's Cyanide Slime. H. Ost and C. Kirschter. (*Zeit. angew. Chem.*, 1905, 1323.)—W. Feld has given the formula $(NH_4)_2Fe.FeCy_6$ to the insoluble cyanide compound contained in this slime.

According to the results of numerous analyses by A. Hand (*Zeit. angew. Chem.*, 1905, p. 1098), this is incorrect. He finds in the slime an insoluble cyanide to which he gives the formula $(NH_4)_2FeCy_4$, with which is sometimes mixed a soluble cyanide of the composition $(NH_4)_4FeCy_6$.

The insoluble compound $(NH_4)_2FeCy_4$ may be considered either as a double salt, $2NH_4Cy + FeCy_2$, or as a ferrocyanide, $(NH_4)_6Fe.(FeCy_6)_2$.

The authors have made a full analysis of the soluble and insoluble constituents of the slime, the results of which strongly confirm Hand's formula for the insoluble cyanide constituent. A. N. C.

The Porosity of Building Materials. H. Seger and E. Cramer. (*Chem. Zeit.*, 1905, xxix., 884.)—Thörner has lately published a method of determining the porosity of building material, and the present authors are therefore led to bring forward the method and apparatus they have been using for some time, for which they claim certain advantages. For these controversial points, reference should be made to the original, but a description of Seger and Cramer's process may be here given.

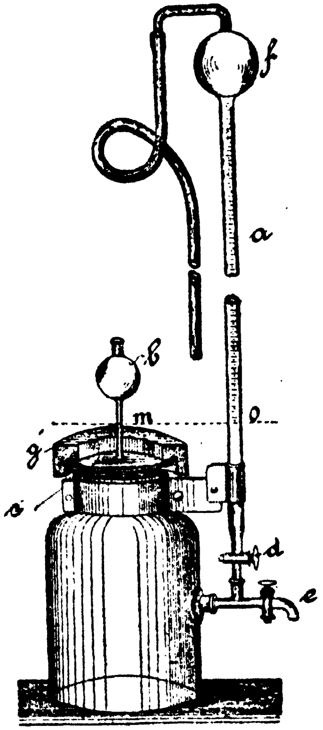


Fig. 1.

The porosity depends upon the specific gravity and a quantity called by the authors "space density" (Raumgewicht), which they define to be the ratio between the weight and the total volume of the body inclusive of the pores.

To determine these quantities, two forms of apparatus have been devised. The first is intended for very accurate work, and is more difficult to use; the second is simpler and easier to manipulate, but is not susceptible of an equal delicacy of measurement.

The first apparatus is shown in Fig. 1. It consists of a wide-necked flask which is connected by a side-piece to a measuring-tube *a*, and a tap *e*. For the determination, the flask is filled with water, the stopper is placed in the neck, and fastened on by means of the metal ring *g*, and the water brought to the zero mark *o* (to which level *m* corresponds on the tube passing through the stopper) by means of the tap *e*. Water is then drawn by suction into the bulb *f*, by means of the flexible rubber tube, until the bulb is about three-quarters full, the tap *d* is turned off, the stopper is removed, and a piece of the material, which has been previously dried and weighed and then saturated with water, is carefully introduced into the flask. The stopper is replaced and fastened down by the ring *g* as before (the authors claim that this method of fastening ensures the stopper always displacing the same volume of water in the neck). The tap *d* is now carefully opened, and the water allowed to run back until it reaches the mark *m*. The volume in the measuring-tube, if the top level is on the scale, is directly read off, and gives the volume of water displaced by the water-saturated piece of material. If some water is still in the bulb, the volume necessary to bring the top level on to the scale is run off by the tap *e* into a graduated cylinder, and the sum of the volumes in the cylinder and measuring-tube taken. The volume run off should be 100 c.c. or so, and a standard 100-c.c. flask can be used in place of the cylinder.

The piece of material is now weighed after removal from the water; the water contained in the pores does not run out.

Suppose, now, that the weight of the dry piece of material is t grams, and its weight, when saturated with water, n grams. Let v be the volume displaced by the water-saturated material. Then the increase in weight due to water taken up $= n - t$, the "space density," $R = \frac{t}{v}$;

the specific gravity, $S = \frac{t}{v - (n - t)}$;

the "pore volume," expressed in hundredths of the volume, $P = \frac{100(n - t)}{v}$;

and the capacity for taking up water W, expressed in hundredths of the weight

$$= \frac{100(n - t)}{t}.$$

This last quantity, W, can be used as an approximate measure of the porosity of the material.

The second form of apparatus is shown in Fig. 2. It consists of a glass cylinder *a*, with a wide flange ground flat, and a side tube and tap *b*. The cover *c* also has a flange ground smooth, so as to afford, with the aid of vaseline, a water-tight junction with the cylinder. The cover is conical, and passes into a tube, which, in turn, is continued to form a funnel. The mark *o* is cut on the tube. This cover is kept in position by a heavy metal ring *e*. The diameter of the cylinder is somewhat less than the lower diameter of the cover, in order to prevent the imprisonment of air bubbles when water is poured in.

In use, the apparatus is first filled to the mark *o*. Water is then withdrawn by the tap *b* until the level is well down in *a*. This water is collected in a weighed beaker. The cover is then removed, and a piece of material, weighed and saturated with water as before, is introduced into *a*. The cover is replaced, and water poured back from the beaker until the mark *o* is again reached. The beaker is then again weighed, and the increase in grams gives the volume displaced by material in c.c. The calculations are made as before.

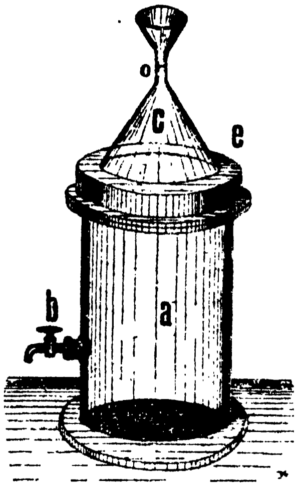


Fig. 2.

The apparatus described can be obtained from the Chemical Laboratory for Clay Industry, Professor Dr. H. Seger and E. Cramer, Berlin, N.W., 21, Dreysestrasse, 4.

E. K. H.

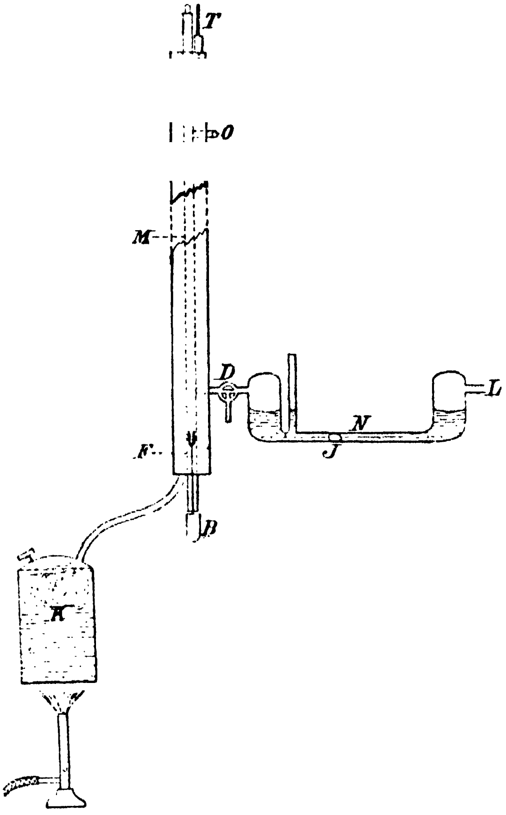
APPARATUS.

Crucibles, Tubes, Combustion-Boats, etc., of Pure Magnesia. (*Chem. Zeit.*, 1905, xxix., 885.)—Experiments carried on in the research laboratory of the Royal Porcelain Manufactory have been successful in producing tubes up to 80 centimetres in length and 7·5 centimetres in diameter, with a wall of 7·5 mm., also crucibles up to 50 centimetres in depth, from pure magnesia.

They withstand temperatures as high as 1,750° C. without any apparent loss or trace of electrolysis: they do not change in shape nor crack. E. K. H.

A New Apparatus for the Determination of Vapour Densities. E. Grimsell. (*Zeits. f. angew. Chem.*, 1905, xviii., 734.)—The principle of this new method is that the weight of a column of vapour of measured length is compared with the weight of a column of air of equal height by means of a sensitive manometer.

The apparatus (see figure) consists of a vertical thin-walled brass tube, *M*, 130 cm. long and 6 mm. wide. Fifteen cm. from the lower end, and at the same distance from the upper end, are short brass tubes at right angles to the long tube; this is surrounded by an outer tube about 5 cm. wide, which serves as a vapour jacket. The upper and lower ends of the narrow tube are brought through the ends of the jacket and made vapour-tight with solder. The short side-tubes go through the wall of the jacket. In the bottom of the vapour jacket is a copper tube, which brings in the vapour from the copper boiler, *K*. The top of the jacket is provided with a little collar, in which a thermometer, *T*, is fixed loosely. The evolution of vapour can be regulated by a flame set under the boiler so that the temperature of the heating vapour is maintained throughout the jacket without letting the vapour escape too much through the upper opening. The condensed vapour runs back into the boiler. The upper end of the inner tube is normally kept closed, and is used only for cleaning purposes. In the lower end of the inner tube fits a stopper with a wooden handle; the top of the stopper is in the shape of a small cup, in which the substance to be vaporized can be placed. The lower side-tube is connected through a three-way tap with a sensitive manometer, *N*; this is filled with coloured water, an air-bubble, *J*, in the horizontal tube acting as index. A small difference in level between the two wide cylindrical columns causes a linear displacement of *J* of fifty times the extent, and the instrument is thus very delicate.



The upper side-tube, which is exactly 1 metre above the lower, is closed by a small metal stopper, *O*, with a fine hole bored through.

The three-way tap, *D*, allows the inner tube to be placed in communication with the manometer, or with the outer air, or to be closed, in which case the manometer is in communication with the outer air.

In working, the three-way tap first closes the inner tube, and the substance is introduced when the temperature has become steady. Almost instantaneously vaporization occurs, and the air is forced out through the upper opening. As soon as this rush of air has ceased (which can be determined by placing a small flame at O), connection is made between the inner tube and the manometer, and the deflection of the air-bubble read off.

If now the area of cross-section of the vapour-containing tube is 1 square cm., the volume of vapour = 100 c.c., and the weight of this column $= 100s_1$.

On the other side of the manometer there is the pressure of the atmosphere, but of this only 100 cm. is balancing the vapour column, as on the side of the vapour there is the atmosphere acting above, O. There is thus a column of air of 1_0 cm. height balancing the vapour column.

The difference in pressure is, therefore, $100\, s_1 - 100\, s_0$, where $s_0 =$ the density of the air.

If the ratio between the diameters of the two wide columns and that of the connecting-tube in the manometer is n, and the index is deflected p mm., then the difference in pressure is $\frac{p}{10n}$ grams.

We have thus the equation $100(s_1 - s_0) = \frac{p}{10n}$, whence $s_1 = s_0 + \frac{p}{1000n}$.

This gives the vapour density at the temperature of the heating vapour, and at the existing barometric pressure. The density at 0° C. and 76 mm. is given by

$$s = s_1 \times \frac{760}{b} \times \frac{\mathrm{T}}{273}$$

$$= \frac{760}{b} \cdot \frac{\mathrm{T}}{273} s_0 + \frac{760}{b} \cdot \frac{\mathrm{T}}{273} \cdot \frac{1}{1000n} \cdot p,$$

$$\text{or } s = \mathrm{C}_1 + \mathrm{C}_2 p,$$

where C_1 and C_2 can be calculated once for all, if barometric variations are neglected.

E. K. H.

Chemical Glassware. P. H. Walker. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 865-875.)—The author has examined eleven different makes of flasks, beakers, etc., and finds that vessels which bear a trade-mark are made of zinc borosilicate glass. Of these, the Wiener Normal glass is the least resistant to reagents, and its properties more nearly resemble the alkali lime silicate glasses; the remainder vary so little in quality that the choice may be reduced to a simple consideration of price. The alkali lime silicate glasses found on the American market are usually of very poor quality, and as they are unmarked there is no means of identifying the different make, and consequently an entirely different make of glass to the one ordered may be received.

W. P. S.

Standard Powders for Analysts.—This is a collection of guaranteed standard samples of foods and drugs in powder form, specially designed for the use of Public Analysts, candidates for the examinations of the Institute of Chemistry, and for students generally. They are put up in a case, in neat form, and will be found very useful when a microscopic examination of a doubtful specimen has to be made. They may be obtained from Southall Bros. and Barclay, Birmingham. W. J. S.

The True Drop-point, and an Apparatus for its Determination.

L. Ubbelohde. (*Zeit. angew. Chem.*, 1905, 1220.)—The determination of the drop-point of fats and oils that have no sharp or characteristic melting point is often a useful test.

The two methods in general use are those of Pohl and Finkener. In the latter the end of a glass rod 3 mm. in diameter is dipped 10 mm. deep twice at three minutes' interval into a melted sample of the fat to be tested, and the principal source of error in the method is the variable ratio of the mass of fat thus obtained on the glass rod to that of the drop which forms from it in the course of the test.

By a series of observations, in which the mass of fat taken on the rod was progressively increased, it is shown clearly that the readings of the drop-point vary least when this ratio is near its maximum, and it is noteworthy that the readings obtained under these conditions agree very closely with those obtained by single observations with the new apparatus about to be described.

This, as shown in the illustration, consists of a thermometer, *a*, to which is fastened a cylindrical metal case, *b*, in which is a small hole, *c*. Into the under part of this metal case fits the glass vessel, *c*. This last is 10 mm. long, 7 mm. wide, and has an opening below 3 mm. wide.

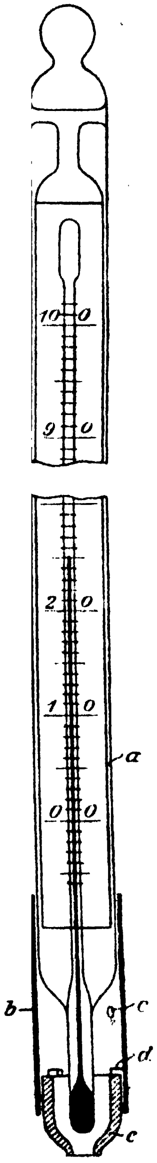
The glass is exactly filled with the fat to be tested, and replaced in the metal case well up to the three stops, *d*. The apparatus is then fastened through a bored cork into a test-tube 4 c.m. wide, and gently heated in a water-bath. The rise in temperature should be at the rate of 1° per minute.

Shortly before the first drop falls the flat surface at the opening below swells out to a convex form. The temperature at the moment the drop falls is the drop-point for that particular substance. As the temperature of the outer surface must be slightly greater than that of the bulb of the thermometer, a small correction of the actual reading is necessary in order to obtain the true drop-point, 0·5° being added for every 1° rise of temperature per minute. Thus:

Drop-point as read	60·5°
Rise of temperature in water-bath ...	1·0° per minute.
Correction	+0·5°
True drop-point	61·0°

This true drop-point is defined as the temperature at which a drop falls by its own weight from a mass of substance that is being uniformly heated, and the amount and weight of which has no influence on the drop, and the author claims that with this apparatus it can be easily and accurately ascertained.

A. N. C.



INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

SCHEME FOR EXAMINATIONS IN TECHNICAL CHEMISTRY.

IN December, 1902, representation was made to the Council that the Institute of Chemistry would materially expand its sphere of usefulness by holding examinations in technical chemistry, and that by so doing it would stimulate the acquisition, by at least a section of the Fellows, of a knowledge and experience which would render them better equipped for positions in chemical industries. The matter was referred to the Council elected in March, 1903, who appointed a Special Committee "to consider the suggestion that the Institute should hold examinations with a view to the promotion of the training of technical chemists."

The outcome of this was that the Committee, after receiving many valuable suggestions, and after giving them full consideration, submitted a report to the Council recommending the establishment of examinations in technical chemistry, together with a scheme largely based on the opinions of recognised leaders of industry. The report was received, and subsequently the scheme, with a series of suggested questions, was widely circulated for criticism. The Committee again received many opinions, the consideration of which led to some modification of the scheme. This was eventually adopted in the form subjoined.

1. The Council have resolved to hold examinations in the industrial applications of chemistry, and to grant certificates in respect thereof.

2. These examinations will be open only to Fellows, and to those Associates who have been registered as such for at least one year.

3. Candidates will be required to produce evidence of practical technical training.

4. The examinations will comprise the following :

   (*a*) The application of well-known chemical and physical laws to industrial operations.
   (*b*) The development, control, and transmission of power and heat.
   (*c*) A working knowledge of operations and plant of which general use is made in industrial works for the treatment and handling of materials, finished products, waste products and effluents, including a practical acquaintance with fittings and stores.
   (*d*) The properties of materials affecting their application to the construction of plant and apparatus in chemical works.
   (*e*) Some ability in interpreting drawings of chemical plant and in making dimensioned rough sketches.
   (*f*) The calculation of working costs, and a general knowledge of the clerical work connected with manufacturing operations.

5. Each candidate will be required to select one important industry in which his knowledge of the above subjects may be tested. Questions which might involve the disclosure of unpublished processes and details of plant in particular works will not be asked. All candidates will be expected to give evidence of a general knowledge of chemical technology. The examiners will take into account original work and special knowledge, but not so as to excuse the candidate from any part of the examination.

NOVEMBER, 1905. Vol. XXX., No. 356.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. C. F. Cross, B.Sc., and C. E. Male were read for the second time; and certificates in favour of Messrs. George Clarke, Jun., F.I.C., analytical chemist, head of the Chemical and Agricultural Department, County Technical Laboratories, Chelmsford; Charles Alexander Hill, B.Sc. (Lond.), F.I.C., director of the laboratories of Messrs. Davy, Hill and Company, 64, Park Street, Southwark, S.E.; and Henry James Horton, 94, Chapelfield Road, Norwich, chemist to Messrs. A. J. Caley and Son, Norwich, were read for the first time.

The President announced that the Council had nominated Dr. H. W. Wiley, Chief Chemist of the United States Department of Agriculture, for election as an Honorary Member of the Society.

The following papers were read: "A Rapid Method for the Determination of Tin in Copper-Tin Alloy," and "Water from the Simplon Tunnel," by Arthur G. Levy, B.Sc.; Notes on (1) "Dika Oil" and (2) "Surin Fat," by J. Lewkowitsch, M.A., Ph.D.; and "The Determination of Oxygen in Copper," by L. Archbutt.

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A RAPID METHOD FOR THE DETERMINATION OF TIN IN COPPER-TIN ALLOYS.

BY ARTHUR GARFIELD LEVY, B.Sc.

(*Read at the meeting, November* 1, 1905.)

IN the analysis of bronzes and similar alloys a good deal of time is required for the determination of tin by the ordinary methods of separation, as copper and lead must first be removed, unless the metal is treated with nitric acid and the tin precipitated as stannic hydroxide. In this case, however, as is well known, the washing out of the copper is generally tedious, and if the quantity of tin exceeds 1 or, at most, 2 per cent. the precipitate obtained is certain to be contaminated with oxides of other metals, the quantities of which must be determined and subtracted from that of the tin dioxide found.

These considerations led to a search being made for another method which should give a direct and accurate determination of the tin in a few hours. The ease with which arsenic in copper is separated by the distillation of its chloride suggested an analogous method being tried for tin. For this purpose the alloy was dissolved in various solutions, such as aqua regia, ferric chloride solution, and potassium cupric chloride solution into which chlorine was led. It was found, however, that distillation from these aqueous solutions was a tedious process, even when a stream of dry hydrogen chloride was led through the retort during the operation; several distillations to dryness, with fresh quantities of hydrochloric acid, were always necessary to remove the tin completely. Consequently, this method was abandoned.

The general method of separating tin, antimony, arsenic, and bismuth from the other members of the second group by heating the metals in a stream of chlorine is well known, though perhaps little used. It is mentioned already in the first edition of Rose's "Lehrbuch der Analytischen Chemie." The same author states, however, that if the method is used to separate copper and tin, a trace of tin remains behind with the copper (Pogg. Anal., 1861, CXII., 169). The method is briefly described in Fresenius ("Quantitative Analysis," vol. ii., 431), Rose's statement being quoted. Menschutkin ("Analytical Chemistry," English edition, 349) mentions the general method as affording an easy means of separation, but does not apply it to the particular case of tin and copper. Lunge ("Chemisch-Technische Untersuchungs Methoden," vol. ii., 285) recommends the decomposition by heating in a stream of chlorine only for white metals rich in tin and antimony. Other works on technical analysis, such as those of Classen and of Brearley and Ibbotson, do not give the method at all. It seemed worth while, then, to see what results could be obtained in this way for copper-tin bronzes. For this purpose the alloy was at first heated in the ordinary double-bulbed, hard glass tubes in a stream of chlorine. It was found, however, that the reaction was so violent that a good deal of spirting took place, so that whilst it seemed an easy matter to drive the whole of the stannic chloride into the receiver, the solution obtained contained considerable quantities of copper and any other metals present. The heavy vapour of the stannic chloride seems to lend itself especially to the carrying forward of particles of the solid or liquid chlorides of other metals. In Lunge's work this difficulty is mentioned in connection with the analysis of white metals rich in lead.

With this method the fault appeared to lie chiefly with the apparatus employed. Consequently, this was modified until the following form was evolved, with which good results have since been obtained: The alloy is placed in a 30-c.c. distillation flask, the side tube of which is bent first up for a short distance, and then down again, so as to allow any drops of liquid projected into the opening of the side tube to drain back into the flask. The entry tube for the chlorine reaches about halfway down the bulb part of the flask; around it, and below the side tube, is wrapped fairly tightly a plug of glass wool, which effectually retains the fine spray of other chlorides carried up by the stannic chloride. The distillation flask is connected by a bent glass tube with two Volhard receivers containing water, into which the stannic chloride distils. It has been found practicable to use rubber stoppers and joints, as, although these perish eventually, they last for a sufficient length of time to render the use of ground-glass joints unnecessary.

In making a determination with this apparatus, $\frac{1}{2}$ gram of the alloy in the form of sawdust or drillings is weighed into the flask, which is then placed in position. It is necessary to use the alloy in a fairly finely divided state, as, if pieces are employed, they become coated with a skin of chloride, which stops the further action of the chlorine completely, until a fairly high temperature is reached, at which this skin melts and a violent reaction takes place, during which chlorides of other metals than tin may be projected into the first receiver in spite of the glass-wool plug.

A fairly rapid stream of chlorine is passed through the apparatus. The chlorine contains hydrogen chloride, derived from its passage through a wash-bottle containing fuming hydrochloric acid; it must be thoroughly dried before it enters the distillation flask. The object of having hydrogen chloride present is to convert any oxide formed from air accidentally present into chloride, which the chlorine alone would not be able to do. After the flask has become filled with chlorine it is gently warmed. The reaction takes place readily, and it is an easy matter to drive the stannic chloride into the receivers. It has been found, however, that it is difficult to sweep out the last traces of the vapour, using only a gas. For this reason, when apparently the whole of the stannic chloride has been distilled off, the heating is stopped, and the flask allowed to cool; the flask is then unstoppered, the glass plug pushed down into the flask by means of the chlorine entry tube, and 10 c.c. of hydrochloric acid are added, after which the stopper is replaced, and the flask again heated in the current of chlorine until the bulk of the liquid has been distilled off. This treatment sweeps out the last traces of stannic chloride into the receivers, and only occupies a few minutes.

The tin in the receivers is precipitated as sulphide, after partial neutralization, if necessary, of the acid liquid by ammonia. The precipitate will, of course, contain any antimony, arsenic, or bismuth present in the alloy. Antimony and, especially, bismuth do not occur in ordinary bronzes in weighable quantities, and hence may generally be neglected; arsenic is removed during the subsequent ignition to oxide. The further treatment of the sulphide obtained depends on whether antimony is especially to be looked for or not. If it is, the thoroughly washed precipitate is transferred to a small weighed dish, and oxidized as usual with nitric acid, the oxides obtained being finally ignited at a dull red heat. Antimony and, if thought necessary, arsenic are then determined as usual, and tin taken by difference. In the case of an ordinary alloy, however, the washed tin sulphide is simply ignited directly, at first very gently, and finally at a fairly bright heat. The trace of antimony present is not affected by this treatment, and may be identified as usual with iron wire and platinum after fusing the stannic oxide. It is well to reignite the latter, as the first weight obtained is occasionally slightly too high if the temperature of ignition was too low.

The results obtained by using this method for bronzes are shown in the following table:

	Percentage of Tin found by		Error.
No.	Usual Method.	Chlorine Method.	Per Cent.
1	11·50	11·47	−0·03
2	14·37	14·32	−0·05
3	9·38	9·29	−0·09
4	9·77	9·72	−0·05
5	11·55	11·58	−0·03
6	10·21	10·13	−0·08
7	9·55	9·50	−0·05
8	0·99	0·98	−0·01

Most of these bronzes contained up to 10 per cent. of lead, together with small quantities of phosphorus and arsenic. One or two contained in addition about 10 per cent. of zinc, the balance being copper.

A brief description of the "usual method" mentioned in the above table may not be out of place here. It consists in dissolving the alloy in aqua regia, precipitating the copper by means of ammonium thiocyanate and sulphur dioxide, and passing hydrogen sulphide into the filtrate from the cuprous thiocyanate after removing the sulphur dioxide by leading a current of air through the heated solution for some time. From the precipitate of mixed sulphides obtained the stannic solution is dissolved out by sodium sulphide solution. The solution is acidified with hydrochloric acid, after which the stannic sulphide is filtered off, washed, redissolved in ammonium sulphide solution to remove sodium salts completely, and finally oxidized with nitric acid, or ignited to oxide as above.

The chlorine method has also been tried on commercial samples of tin, with fairly good results:

	Percentage of Tin found by		Error.
No.	Difference.	Chlorine Method.	Per Cent.
1	99·88	99·66	−0·22
2	99·35	99·38	+0·03
3	96·73	96·62	−0·11

A determination of tin by the chlorine method can be finished in about five hours.

The method, therefore, is of value where a rapid and accurate check on the percentage of tin is wanted. If a complete analysis of the alloy is to be made, it has not very much to recommend it, although even in this case several hours can be saved by determining the tin on a separate portion treated as above. Naturally, the use of chlorine tends to make it somewhat disagreeable for the operator.

The work described was carried out in the laboratory of Mr. Bertram Blount, to whom I am very much indebted for permission to publish the results.

Discussion.

The President (Mr. Bevan) having invited discussion,

Mr. Blount said that the history of Mr. Levy's work on this subject turned on the fact, long ago observed and generally known, that stannic chloride was volatile. In analysing samples of ordinary commercial tin, he (Mr. Blount) had sometimes found it convenient to get rid of the tin by repeatedly evaporating with hydrochloric acid the solution of stannic chloride obtained by dissolving the metal in aqua regia. That had been found effective. He then conceived the idea of distilling over the tin from alloys in the same way, and that was the genesis of Mr. Levy's work. It was found, however, that the volatilization of the stannic chloride in that manner was impracticably slow, and so the more conventional process was adopted of converting it into chloride and getting over the chloride at a higher temperature than that of distillation from an aqueous solution; and, the difficulties connected with the apparatus having been solved as Mr. Levy had described, a really good working method was obtained for the determination of tin in alloys that were not too complicated. Ordinary gun-metal, for instance, which was somewhat troublesome in the ordinary way of analysis, could be readily dealt with. The determination of the tin after distillation was much simpler than had been previously supposed, no greater precautions being necessary in dealing with the tin sulphide than were necessary with zinc sulphide.

Mr. **Archbutt** said that the use of chlorine was, of course, the great objection to a process of this sort, and probably if it were an odd sample to be dealt with, and especially if it had to be analysed completely, such a method would not be advisable; but if there were a number of analyses to be made regularly, and it was known how much tin was usually present, and that the samples were fairly free from antimony and other volatile metals, the method should prove most useful. With regard to the prevention of splashing, he thought the glass-wool plug might be dispensed with if the neck of the flask were made very long, say 12 inches. This expedient, which he had found very useful in determining ammonia in water, and also in distilling arsenic from copper, enabled boiling or distillation to be carried on very rapidly without any loss through splashing. He should like to hear more details than Mr. Levy had given of the process used when antimony was present. Most of these alloys, in his experience, contained small quantities of antimony, the influence of which was somewhat important. He had himself found, though he had not gone into the matter very closely, that the ordinary nitric acid method of separating tin from other metals, such as copper, when the proportion of tin was more than 10 or 12 per cent., was by no means so inaccurate as the books stated. Very good results could be obtained by dissolving the metal in nitric acid of 1·2 specific gravity, evaporating to a small bulk, diluting, filtering, and washing the tin oxide, which was far from being so impure as to invalidate the use of the method. It contained nearly all the antimony present, and if, after being weighed, the mixed oxides of the tin and antimony were fused with sulphur and sodium carbonate, dissolved in water, and treated with a large excess of strong oxalic acid solution, the antimony was precipitated. For foundry analyses he had found that method very satisfactory, and with a little management it was possible to get the tin oxide filtered and washed within a reasonable time. He did not, however, doubt that the method now described was a great improvement.

Mr. **Bodmer** asked what method had been found most convenient for generating the chlorine, and how the gas was obtained in a perfectly dry condition.

Mr. **Chapman** said that Vortmann and Metzl had recently published a method for the separation of antimony from tin, in which the mixed sulphides were dissolved in hydrochloric acid, the greater part of the acid being then neutralized and a certain quantity of syrupy phosphoric acid added. The authors claimed that the antimony could then be quantitatively precipitated by sulphuretted hydrogen. One advantage of such a method would be the ease with which the tin could be determined in the filtrate. The results of the specimen analyses that were given seemed to be very satisfactory, and if the statements made with regard to it were correct the method would be a good one.

Mr. RICHARDS said that some years ago he had had occasion to examine certain dental alloys containing platinum, gold, silver, and tin, and had determined the tin with good results by a process somewhat similar to that used by Mr. Levy. The formation of tin chloride was complete in every case, and the use of a little glass-wool effectually prevented any trouble from the passing over of other chlorides. The separation of the other metals afterwards was effected without difficulty.

The PRESIDENT asked how Mr. Levy ascertained that sufficient antimony was present in the distillate to make its determination worth while.

Mr. LEVY, in reply, said that alloys of this kind were generally made from carefully chosen metals, and he thought that in most cases the tin used would not contain more than 0·1 per cent. of antimony at the outside, so that, with alloys such as those he had referred to in the paper, it would be scarcely worth while to actually determine the antimony. If any fairly large proportion of antimony were present, a sufficient indication of it would be obtained in the course of the operations he had described, and in that event one would have to repeat the determination and treat the antimony more carefully. The usual method of testing for antimony was to fuse the stannic oxide again with a small quantity of soda, dissolve the melt in a small excess of hydrochloric acid, and treat in a platinum dish with a piece of iron wire. The merest trace of antimony would thus be shown after heating for a quarter of an hour on the water-bath. The tin precipitates were treated in that way as a matter of routine. He had seen the paper to which Mr. Chapman had referred, but had never tried the process. The results given, however, appeared to be excellent. He had seen a good many results obtained by the oxalic acid method, and some of them were very good, but he had never been able himself to get very good results with it, as far as the determination of antimony was concerned. As a rule, the tin was taken by difference. The method he preferred for the separation of considerable quantities of tin and antimony was that of Rose. It was certainly a long and cumbrous method, but when carefully carried out it yielded very good results, both for small quantities of tin in presence of large quantities of antimony, and *vice versâ*. The chlorine, for a distillation process like this, where not very much was required, was conveniently generated from hydrochloric acid and ordinary granulated manganese dioxide in a 16 or 20 ounce flask. The drying was effected simply by leading the chlorine through sulphuric acid wash-bottles and tubes. It was important to see that the sulphuric acid was really fresh and good. He had lately used a Streatfeild drying-tube. He did not think that the liability of other chlorides to be carried over was a question of ordinary splashing. The stannic chloride vapour seemed to lend itself specially to the carrying over of the chlorides of other metals in a finely divided form, probably because, being heavy, it did not readily allow any dust that got into it to fall down, so that the use of something which, like glass-wool, acted mechanically as a sort of sieve, was necessary. He thought that the most troublesome part of the nitric acid method was the filtering off of the stannic hydroxide or stannic oxide. The precipitate was very troublesome to wash if it was at all large in quantity, and perhaps the correctness of the results might be attributed to a sort of compensation of errors. If, however, the alloy contained, say, 15 per cent. of tin and only about 0·1 or 0·2 per cent. of lead, it did not seem quite fair, as far as his experience went, to throw a fairly considerable percentage error on the lead.

The PRESIDENT said that he had found the simplest way of generating chlorine to be to drop hydrochloric acid on to permanganate of potash. The production of chlorine in that way could be regulated with the utmost nicety.

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WATER FROM THE SIMPLON TUNNEL.

By ARTHUR GARFIELD LEVY, B.Sc.

A SAMPLE of water from one of the hot springs encountered during the construction of the Simplon tunnel between Switzerland and Italy, obtained from Mr. Francis Fox, the well-known engineer, was recently examined by the author in the laboratory of Mr. Bertram Blount. The spring was situated at a point between 9·1 and 9·4 kiloms. from the Italian end of the tunnel. It discharged water at the rate of 230 litres per second, the temperature of the water being 45·9° C.

The water itself was clear, colourless, and devoid of smell, and had a somewhat saline taste. It was free from organic matter and from chlorine, the total absence of the latter being rather remarkable.

The mineral constituents present are as follows:

	Grams per Litre.	Grains per Gall.
Silica	0·0102	0·71
Alumina and ferric oxide	0·0022	0·16
Calcium oxide	0·4910	34·37
Strontium oxide	0·0030	0·21
Magnesium oxide	0·0655	4·59
Potassium oxide	0·0050	0·35
Sodium oxide	0·0086	0·60
Carbon dioxide (combined)	0·0267	1·87
Sulphur trioxide	0·8000	56·00
	1·4122	98·86
Combined water and loss	0·1098	7·68
Total solids	1·5220	106·54

A trace of lithium was also present. No barium, cæsium, or rubidium could be detected by the spectroscope.

From the above analysis the salts probably present in the water are calculated to be as follows:

	Grams per Litre.	Grains per Gall.
Calcium carbonate	0·0607	4·25
Strontium sulphate	0·0053	0·37
Calcium sulphate	1·1099	77·69
Magnesium sulphate	0·1965	13·77
Potassium sulphate	0·0092	0·65
Sodium sulphate	0·0170	1·27
Silica	0·0102	0·71
Alumina and ferric oxide	0·0022	0·16
	1·4110	98·77

The water is very rich in calcium sulphate, besides which there are present a considerable quantity of magnesium sulphate and small amounts of other salts.

An examination of the gases expelled by boiling the water showed it to contain only the ordinary constituents of the atmosphere, the quantities found being as follows:

	C.C. Dry Gas per Litre at 0°C. and 760 Mm.	Percentage on Total Gas.
Carbon dioxide	2·0	9·6
Oxygen	6·1	29·4
Nitrogen	12·3	59·1
Argon	0·4	1·9
Total	20·8	100·0

The gases are evidently due to absorption from the air.

To Mr. Francis Fox for providing the opportunity to examine a water of considerable chemical interest, and to Professor Herbert Jackson for kindly making the necessary spectroscopic tests, I wish to offer my acknowledgments and thanks.

DISCUSSION.

Mr. BLOUNT said that, as this water was possibly entirely plutonic, having never seen the surface of the globe, something out of the common had been looked for in its results. The only interesting point, however, was the complete absence of chlorine. He had never met with a natural water before that was completely destitute of chlorine; it was unique.

The PRESIDENT (Mr. Bevan) inquired to what extent the water had been concentrated before the test for chlorine was made, and also whether the water had been tested for barium.

Mr. W. T. BURGESS remarked that, as a rule, the quantity of chlorine in natural water became less as the distance from the sea was greater. In France, for instance, it was possible to get rain-waters much freer from chlorine than in England, and possibly the distance from the sea which the water had to travel before being deposited as snow and subsequently melted might have some bearing on its freedom from chlorine.

Mr. FAIRLEY said that in Asia and in America it was possible to get much farther from the sea than anywhere in Europe, but the Great Salt Lake of Utah and some of the lakes of Central Asia were exceedingly saline.

Mr. Blount suggested that possibly those lakes were themselves the remains of ancient seas.

Mr. Levy, in reply, said that chlorine was only looked for in the water as received, without any concentration. The tests, however, were allowed to stand for a very long time, in one case (using 250 c.c. of water) for four or five days. It was a difficult water to concentrate, because, on account of the large quantity of calcium sulphate present, a crust separated out. Professor Herbert Jackson had kindly made a spectroscopic examination of the sample, and had found in it a trace of lithium, but no barium or cæsium. Probably about 200 miles was the distance of the source of this water from the sea.* No doubt the altitude would also have to be taken into account. At the same time, to have attained so high a temperature in such a region, it must have travelled a considerable distance underground, and it was rather remarkable that it did not take up chlorine somewhere on its way.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Determination of Higher Alcohols in Spirits. E. Beckmann. (*Zeit. Nahr.-Genuss.*, 1905, x., 143-152.)—The following method is a modification of the one described by the author some years ago (*cf.* Analyst, 1902, xxvii., 88). The spirit is diluted with water until it contains not more than 20 per cent. of alcohol by volume. Fifty c.c. of this solution are then shaken out with four successive quantities of carbon tetrachloride, using 50 c.c. of the latter each time and shaking vigorously for a few seconds. The united carbon tetrachloride extracts are washed twice with about 20 c.c. of water and then treated in a stoppered flask with 2 grams of potassium hydrogen sulphate and 1 gram of sodium nitrite. After shaking for a few minutes, the mixture is transferred to a separating-funnel, the carbon tetrachloride is drawn off and the residue extracted twice with a little carbon tetrachloride. The whole of the solution is now shaken with about 20 c.c. of saturated sodium-hydrogen-carbonate solution to remove excess of nitrous acid, and separated. The carbon tetrachloride is then carefully mixed with about 75 c.c. of concentrated sulphuric acid previously placed in a separating-funnel, and well shaken. The mixture is finally run on to about 150 grams of crushed ice and the resulting solution titrated with permanganate solution. To obtain a sharp end-point, it is advisable to add an excess (about 20 per cent.) of the permanganate and to titrate back with ferrous ammonium sulphate solution. W. P. S.

Detection of Methyl Alcohol in Liquids containing Ethyl Alcohol. J. Kahn. (*Merck's Report*, September, 1905, 271; through *Pharm. Jour.*, 1905, lxxv., 387.)—The test proposed depends on the oxidation of the mixed alcohols to the corresponding aldehydes by the action of heated copper, any formaldehyde formed being detected by the appearance of a violet-blue coloration when tested with milk and commercial sulphuric acid. After dilution, the solution to be tested is warmed in a large test-tube, into which a red-hot copper spiral is dipped several times. Five c.c. of milk and a trace of ferric chloride are now added, and the mixture carefully poured on the surface of concentrated sulphuric acid contained in another test-tube. If methyl alcohol is present in the original solution, the formaldehyde produced by the oxidation will cause a violet-blue coloration to appear at the junction of the two liquids. The milk used must, of course, be free from formaldehyde.

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* The actual distance to Genoa, the nearest point on the Mediterranean, is 200 kilos (about 125 miles). —A.G.L.

W. P. S.

The Characteristics of Camel's Butter. J. Vamvakas. (*Ann. de Chim. anal.*, 1905, x., 350.)—Butter prepared in the laboratory from camel's milk was of firm consistency and had a grayish-white colour and a peculiar odour. Its physical and chemical values were as follows: Melting-point, 38° C.; melting-point of fatty acids, 47° C.; volatile fatty acids, 8·6 per cent.; insoluble fatty acids, 88·29 per cent.; saponification value, 208; iodine value, 55·1; and oleo-refractometer reading, 20.

C. A. M.

Detection of Small Quantities of Sesame Oil in Butter, etc. H. Sprinkmeyer and H. Wagner. (*Zeit. Nahr.-Genuss.*, 1905, x., 347-353.)—In the method proposed, the substances which give the colour-reaction with Baudouin's test are concentrated in a fraction of the fat by extracting the latter with acetic acid. About 100 grams of the melted and filtered butter-fat are twice extracted with from 20 to 30 c.c. of glacial acetic acid at a temperature of 60° C. After separation, the acid layers are evaporated, and the residue obtained tested as usual. As many samples of butter contain colouring substances which give a red tint when treated with hydrochloric acid, it is better to remove these substances before applying the test. This may be done by adding 10 c.c. of alcohol and 5 c.c. of saturated barium hydroxide solution to the acetic acid residue, and again evaporating to dryness. The residue is then extracted several times with light petroleum, which dissolves the substances yielding Baudouin's reaction, the colouring matter of the butter remaining insoluble in the barium soaps. The residue obtained on evaporating the petroleum extracts is then tested with the reagent. As little as 0·1 per cent. of sesame oil in butter may be detected by proceeding as above.

W. P. S.

Test for the Purity of Cocoanut Oil. E. Milliau. (*Compt. rend.*, 1905, cxl., 1702; through *Pharm. Journ.*, 1905, lxxv., 361.)—Four c.c. of the fat are mixed in a test-tube with 2 c.c. of a solution of phloroglucinol in ether; 2 c.c. of a solution of resorcinol in benzene are then added, and the tube and its contents cooled to a temperature of 10° C. After adding 4 c.c. of nitric acid, of specific gravity 1·36, the tube is closed with an indiarubber stopper (washed with the same acid) and shaken for five seconds. Pure cocoanut oil remains unaltered, negligible traces of impurities imparting a scarcely perceptible rose tint to the mixture, whilst the presence of any seed oil, such as cotton, sesame, arachis, etc., is at once indicated by a distinct currant-red coloration, even if only 5 per cent. of the adulterant be present. The colorations which appear later are to be disregarded. W. P. S.

The Composition of Chestnut Flour. P. Comte. (*Journ. Pharm. Chim.*, 1905, xxii., 200-210.)—Flour obtained from the dried chestnut is used by the peasants throughout Corsica in place of cereal flours. Five samples from different parts of the island, examined by the author, had the following composition : Moisture, 11·20 to 12·46 ; nitrogenous substances, 5·82 to 6·60 ; fat, 2·65 to 2·85 ; sugars and starch, 72·60 to 73·83 ; cellulose, 2·90 to 3·96 ; and ash, 2·34 to 2·50 per cent. The mineral matter contained from 32 to 36 per cent. of phosphoric acid, and from 8 to 12 per cent. of silica, derived almost entirely from the mill-stones. Manganese was also present. A superior quality of the flour prepared from hand-sorted and cleansed chestnuts was almost free from the brown particles and hard lumps found in the ordinary grades, and had the following composition : Moisture, 17·90 ; nitrogenous substances, 7·46 ; fat, 2·72 ; sugars and starch, 68·30 ; cellulose, 1·52 ; and ash, 2·10 per cent. The flour is but little adulterated in Corsica owing to its low price (0·2 to 0·25 franc per kgm.), though powdered acorn flour is sometimes added. Chestnut flour is very refractory to the action of yeast, and therefore does not make good bread. C. A. M.

Investigation of Mace. Utz. (*Chem. Zeit.*, 1905, xxix., 988.)—Pritchard's method of distinguishing true Banda mace from the so-called "Bombay" mace by means of 1 per cent. caustic soda solution has been investigated by the author.

The result of the experiments was that with Banda mace 1 per cent. caustic soda gave either no colouring or only a very slight yellow, and this tint was not deepened by warming. In the case of Bombay mace there was obtained in the cold a yellow-red colour, which appeared rather more quickly on gently heating. The test is clearer and more delicate if strips of filter-paper are used to absorb some of the liquid from the mace and alkali, and dried. The Banda mace gives completely colourless strips, whilst those from the Bombay mace are deep orange. An addition of 5 per cent. of Bombay mace can be easily detected by this means.

Another method of using this test is to examine the alkaline solutions spectroscopically. Banda mace does not affect the spectrum, whilst Bombay mace shows a broad absorption-band near the D line.

In place of caustic soda, a diluted Nessler solution (1 : 1) may be used, with filter-paper strips. The colour then obtained with Bombay mace is raspberry red.

The permanency of the colour on the strips is being investigated. E. K. H.

Determination of Citral in Lemon Oil. G. Romeo. (*Chemist and Druggist*, 1905, lxvii., 408).—The method proposed is based on the fact that when citral is absorbed by potassium sulphite or sodium hydrogen sulphite, with sufficient excess of the hydrogen sulphite for the solution to remain acid after the absorption, the acidity is reduced in the proportion of 3 molecules for each molecule of citral present. The sulphite solution employed is prepared by dissolving 400 grams of crystallized sodium sulphite in 1 litre of water, and adding enough saturated sodium hydrogen sulphite solution to make each 25 c.c. sufficiently acid to neutralize 20 c.c. of $\frac{N}{2}$ potassium hydroxide solution. Five c.c. of the lemon oil are heated with a sufficiency of this solution in a closed flask for three hours on the water-bath, and the residual acid then titrated. Theoretically, 152 grams (1 mol.) of citral should neutralize 3,000 c.c. (3 mols.) of N acid. (In three experiments the actual results were: 2,955 c.c., 2,957·7 c.c., and 2,962·7 c.c.) One c.c. of N acid therefore corresponds with 0·05066 gram of citral, and the amount of citral in the 5 c.c. taken is a matter of simple calculation. In four samples of oil the author found 3·64, 4·48, 3·54, and 3·80 per cent. of citral, whilst an artificial mixture containing 4·98 per cent. yielded 4·86 per cent. These figures corroborate the views held by many analysts that the 7 per cent. citral value is too high. W. P. S.

On the Resins of Scammony. P. Guigues. (*Journ. Pharm. Chim.*, 1905, xxii., 241.)—It is commonly accepted that scammony resin is soluble in ether, but the author finds that certain scammony roots occurring in commerce contain two resins, one of which is soluble and the other insoluble in ether. The insoluble resin, however, is soluble in an ethereal solution of the soluble resin, but is precipitated on the addition of an excess of ether. The scammony resins prepared some five or six years ago were completely soluble, and this is to be attributed to their having been obtained from roots derived from a very limited area, whereas, owing to the exhaustion of the former sources of supply, they are now derived from other countries and presumably other species of plants. Resins actually in commerce contain as much as 50 per cent. of brown resins insoluble in ether and 25 per cent. of white resins. The much greater commercial value of jalap resin is opposed to the conclusion that the latter is present as an adulterant in these resins, and the author's explanation is that the original roots belonged to a species intermediate between *Convolvulus scammonia* and *C. turpethum*, or were mixtures of the two, or that they had undergone some alteration in the course of drying. Some samples of Aleppo scammony root, examined by the author, were found to yield 79 per cent. of resin soluble in alcohol, whereas other chemists had found from 46 to 77 per cent. soluble in ether. In four other samples extracted from the roots by different methods the insoluble portion varied from 9 to 14 per cent. A portion of one of these resins taken up with alcohol, decolorized, and filtered, had an optical rotation of $\alpha_D = -20^\circ\ 43'$ and a refractive index (in 4 per cent. solution) $N_D = 1{\cdot}3659$, whilst the corresponding figures obtained with a decolorized ethereal solution of another portion of the resin were $\alpha_D = -21^\circ\ 43'$ and $N_D = 1{\cdot}3659$. C. A. M.

Tests for distinguishing between Nicotine, Conine, and Sparteine. Reichard. (*Pharm. Centr.*, xlv., 300; through *Pharm. Journ.*, 1905, lxxv., 333.)—Nicotine and conine may be distinguished by means of a reagent made by moistening a little cuprous chloride with water, and heating until the mass assumes a bright green colour. A small quantity of this reagent is mixed with a drop of either alkaloid and a drop of hydrochloric acid. With nicotine, a violet-blue colour is at once formed; with conine, a bright green solution is produced; with sparteine no immediate reaction occurs. If a few drops of a solution of α-nitroso-β-naphthol be evaporated to dryness, and the residue treated with a trace of these bases, a yellow-brown tint is obtained in the case of nicotine and a fine dark green with conine. Ammonium molybdate in sulphuric acid gives a yellowish-green with nicotine, but no reaction with conine or sparteine. On adding ammonium persulphate to the mixture nicotine gives a purple colour, conine and sparteine an intense yellow. Another colour-reaction is obtained with ferric thiocyanate. A mixture of solutions of ferric chloride and potassium thiocyanate is evaporated in a thin film. To the residue thus obtained a trace of nicotine, conine, or sparteine is added, and then a little water; sparteine gives a violet-blue or reddish-violet tint; nicotine and conine yield a green colour. These colours are permanent on drying. By substituting potassium ferrocyanide for the thiocyanate, and proceeding as before, the blue colour which remains after drying is changed to brown by conine, to bright green by nicotine, and to pale violet by sparteine. On treating the dried spots with a drop of potassium thiocyanate, those of nicotine and conine show no change, whilst the sparteine spot becomes pale blue, passing to deep blue on drying.

W. P. S.

Determination of Phosphorus in Phosphorized Oil. H. Enell. (*Pharm. Zeit.*, 1905, l., 601; through *Zeit. Nahr.-Genuss.*, 1905, x., 355.)—The method is based on the determination of the acidity of the oil before and after oxidation with iodine. A weighed portion of about 1 gram of the oil is placed in a stoppered flask and dissolved in a mixture of 10 c.c. of alcohol and 20 c.c. of ether. Twelve c.c. of $\frac{N}{10}$ iodine solution are added, the whole is well shaken for three minutes, and the excess of iodine titrated back with $\frac{N}{10}$ thiosulphate solution. A little phenolphthalein is now added, and the acidity titrated with $\frac{N}{10}$ sodium hydroxide solution. The titration should be continued until the pink coloration no longer disappears on shaking the mixture. The original acidity of the oil is now determined on a fresh quantity of about 1 gram, dissolved in the above proportions of alcohol and ether. The difference in the acidity before and after the treatment with iodine gives the quantity of free phosphorus in the oil. 0·01 gram of phosphorus is equivalent to 16·12 c.c. of $\frac{N}{10}$ sodium hydroxide solution. W. P. S.

The Action of Arrhenal (Sodium Methyl Arsenate) on Alkaloids. Vitali. (*Boll. Chim. Farm.*, 1905, No. 7; *Journ. Pharm. Chim.*, 1905, xxii., 227, 228.)—The formation of an insoluble precipitate on adding arrhenal to the solution of the salt of an alkaloid has usually been attributed to the alkaline reaction of the reagent, but, according to the author, the reaction is more complicated. With strychnine sulphate, for instance, sodium sulphate and the methyl arsenate of the alkaloid are formed, and it is this latter which is precipitated. Other alkaloids also yield insoluble methyl arsenates, many of which are in the form of microscopic crystals, the appearance of which might afford a means of identifying the alkaloids. C. A. M.

ORGANIC ANALYSIS.

The Determination of Acetaldehyde. Seyewetz and Bardin. (*Bull. Soc. Chim.*, 1905, xxxiii., 1000-1002).—The solution of aldehyde should be diluted, so that its strength does not exceed 7 or 8 per cent. Ten c.c. of the liquid are mixed with about 40 c.c. of a 10 per cent. solution of anhydrous sodium sulphite and 1 drop of a 0·2 per cent. alcoholic solution of phenolphthalein, and titrated with standard sulphuric acid, the amount of aldehyde being calculated according to the equation—

$$2Na_2SO_3 + 2CH_3.COH + H_2SO_4 = (HNaSO_3 + CH_3.COH)_2 + Na_2SO_4.$$

In order to prevent loss of aldehyde from volatilization the temperature should be kept at 4° to 5° C. during the dilution and addition of the sulphite. Alcohol, paraldehyde, or acetal, have no appreciable influence on the results, but acetic acid when present must be determined separately. Trioxymethylene is converted into formaldehyde by sodium sulphite, and can then be determined in the same way as formaldehyde itself. The method is stated to give good results with very dilute solutions of aldehyde (1 : 3000). C. A. M.

The Detection and Estimation of Ketones, and the Differentiation of Acetone from other Ketones. M. Kutscheroff. (*Zeit. anal. Chem.*, 1905, xliv., 622-625.)—On applying Rosenthaler's reaction (ANALYST, xxx., 247) to the detection of ketone oil (chiefly methyl-ethyl-ketone), which is used in the proportion of 1 per cent. for "denaturing" spirit, the author was unable to obtain satisfactory results, since vanillin in the presence of strong hydrochloric acid gave either no coloration at all or only a faint one, appearing after hours or even days. Similar results were obtained with certain other ketones, such as methyl-propyl ketone, ethyl-butyl ketone, and acetone. The substitution of concentrated sulphuric acid (specific gravity 1·84) for hydrochloric acid, however, proved satisfactory, and also enabled acetone to be distinguished from other ketones. A solution of 0·3 gram of vanillin in 5 c.c. of rectified spirit gives only a faint yellow coloration with 1 c.c. of sulphuric acid, but if about 1 per cent. of a ketone be present, a bright coloration is immediately produced, which is carmine red in the case of acetone and blue in the case of other ketones, with sometimes a greenish shade (methyl-butyl ketone), which soon disappears. The intensity of the blue coloration increases on standing. The carmine red colour obtained with acetone changes to lemon yellow on the addition of water, but the blue colour given by the higher ketones is not affected by water. If, however, a dilute solution of sodium or potassium hydroxide be added to the diluted liquids, the yellow acetone solution becomes an intense orange red, whilst the solutions of the other ketones are decolorized, or, rather, changed to a faint yellow.

The sensitiveness of the vanillin reaction is very great, and the test is capable of detecting mere traces of ketones. Hence, with the aid of a colour scale prepared with a ketone solution of known strength, quantitative colorimetric determinations can be made. A freshly prepared solution of 15 grams of vanillin in 100 c.c. of concentrated spirit (as free as possible from aldehyde) has proved the most suitable strength in the author's hands. Of this solution, 2 c.c. should be added to 3 c.c. of the solution under examination, and then 1 c.c. of sulphuric acid. When the ketone amounts to 0·1 to 1 per cent., the coloration is immediately produced, but with smaller quantities (0·1 to 0·01 per cent.) it only occurs after some time, and the solutions should not be compared until after the lapse of ten to fifteen minutes, or longer.

For the detection of acetone in the presence of other ketones, the coloured solution obtained in the reaction should be diluted with water, which changes the colour to green, the carmine red of the acetone being changed to yellow. As a corroborative test, the green solution is treated with a 25 per cent. solution of sodium hydroxide, when it becomes orange red in the presence of acetone. The reaction is so sensitive that it will detect 0·01 per cent. of acetone out of a total amount of 0·1 per cent. of ketones. In like manner very small quantities of higher ketones can be identified in the presence of acetone.

As regards the influence of other substances present as impurities in spirit, amyl alcohol and furfural have no disturbing influence. Acetaldehyde, when present in quantity (1 per cent.), produces a coloration similar to that given by higher ketones, but when present in the usual quantity of 0·1 gram per litre, can be neglected. When, however, it reaches the maximum of 0·3 gram per litre observed in crude spirit, it should be expelled by boiling before the vanillin test is made. On distilling the liquid to half its volume, almost the whole of the ketones of higher boiling-point will be found in the residue, whilst about half of any methyl-ethyl ketone present will have passed over with the alcohol. The amount of aldehyde left in the residue is then too small to have any influence upon the test. C. A. M.

Quantitative Hydrolysis of Saccharose, Maltose, Lactose, and Raffinose. B. Pfyl and B. Linne. (*Zeit. Nahr.-Genuss.*, 1905, x., 104-110.)—Under the following conditions of pressure, temperature, acidity and time, these sugars may be quantitatively hydrolysed without the products undergoing any change. The apparatus employed consists of a bronze pressure cylinder, about 30 cm. high, and of 3·2 cm. internal and 4 cm. external diameter. The cylinder is provided with a screw head carrying a valve-chamber, which is fitted with a manometer and a side-tube. By connecting this side-tube with a carbon dioxide cylinder, any desired pressure up to fifty atmospheres may be obtained. Both screw-joints are made tight by lead packing. Inside the pressure-cylinder is placed a thick walled glass tube, constricted at the top and provided with a mark so that an exact volume of solution may be used each time. During the hydrolysis the cylinder is suspended in a glycerol bath heated to the required temperature.

It is impossible to completely hydrolyse saccharose in the presence of inorganic or organic acids without destroying some of the inversion products, but in the presence of carbon dioxide at a pressure of 20 atmospheres and at a temperature of 125° C., saccharose is quantitatively hydrolysed in two and three-quarter hours. The invert sugar formed remains unchanged after a further heating for thirty minutes. Experiments show that neither dextrin, maltose, nor lactose undergoes any change under these conditions.

When subjected for three hours to a pressure of 20 atmospheres and a temperature of 125° C., raffinose is completely hydrolysed into melibiose and lævulose.

Maltose and lactose are best hydrolysed in a 2 per cent. aqueous solution of benzene-sulphonic acid, kept at a temperature of 110° C. for two and a half hours; variation of the pressure from 0 to 50 atmospheres produces no effect on the completeness of the hydrolysis. If saccharose, or invert sugar, be subjected to these conditions, about 17 per cent. of it is destroyed, and this proportion is independent of the concentration of the sugar solution or intensity of the pressure.

The purity of various sugars may be determined by means of the above-described methods, the latter also further serving for the determination of sugars in the presence of each other, as, for instance, saccharose in honey, condensed milk, etc.

W. P. S.

Examination of Linseed Oil used in the Manufacture of Lacs, Linoleum, etc. C. Niegemann. (*Chem. Zeit.*, 1905, xxix., 898.)—Linseed oil, suitable for these purposes, should not form flocks when quickly heated to boiling in a test-tube. Recently oils have been introduced under the title of "lac linseed oil" (Lackleinöl) which apparently exhibit this characteristic, but are still not suitable. In the case of this class of oil, however, the boiled sample showed a turbidity after several days' standing.

Only those oils should be used which give no deposit after standing for a year or so, and on being boiled acquire a clear greenish colour, and show no turbidity on standing for a month in an open test-tube.

E. K. H.

The Estimation of Sulphur in Liquid Fuel and Petroleum. A. Goetzl., (*Zeit. angew. Chem.*, 1905, xviii., 1528.)—To 2 to 3 grams of the liquid fuel in a roomy platinum crucible 4 c.c. of fuming nitric acid are added, and the crucible covered by a watch-glass and allowed to stand for some time, preferably overnight. The crucible is then cautiously shaken, and when no further reaction takes place, even with gentle warming, the contents are cautiously evaporated to dryness on the water-bath.

To the brown aromatic residue, 6 to 8 grams of a mixture of sulphur, silica-free soda and pure nitre, in the proportions of 5 to 1, are added and the mixture heated to incipient melting on the water-bath. After stirring well with a platinum wire, and covering again with more of the soda-nitre mixture, the crucible is heated under the hood directly over a rose-burner. Under these conditions, the combustion takes place very quietly, and when this is complete, the white mass is dissolved in hot water, the solution acidified with hydrochloric acid, and the sulphate precipitated with barium chloride.

The results are very accurate.

Sulphur can be estimated in petroleum by the same method, but it is necessary to use some 10 grams of the oil, and the various operations are rather more troublesome.

A. N. C.

The Determination of Carbon, Hydrogen, and Nitrogen in Cyanides. J. A. Muller. (*Bull. Soc. Chim.*, 1905, xxxiii., 951-953.)—The combustion of the most refractory metallic cyanides is readily effected by the use of an oxidizing mixture of 83 parts of potassium bichromate (previously fused and powdered) and 13 parts of lead chromate (*Ann. Chim. Phys.*, xxii., 141). The combustion tube used by the author is of Jena glass, 87 cm. in length by 15 mm. in internal diameter. Within it is placed a column of copper oxide 26 cm. in length, which is kept in position by rolled strips of platinum-foil at one end and by a roll of copper-wire gauze at the other. After each combustion this copper is reduced by passing a slow current of hydrogen through the tube, which is only heated at the end for a distance of about 10 to 12 cm.

The determinations of carbon and hydrogen are made with a slow current of air during the combustion, whilst the tube is subsequently swept out with a more rapid current for about an hour. The columns of copper and copper oxide are first heated to redness, and about a litre and a half of air are passed through the tube during the whole operation, which takes about one hour and fifty minutes. The semicircular boat in which the substance is introduced is 117 mm. in length by 12 mm. in width, and 6·5 mm. in depth. The material is covered with the dry combustion mixture, of which nearly sufficient to fill the boat is introduced.

In determining the nitrogen, the part of the tube placed in the furnace is progressively heated to redness, while a current of carbon dioxide, supplied under a pressure of about a metre of water, is meanwhile passed through it. The tube is then cooled to a little beyond the column of copper oxide, the boat charged with the substance and combustion mixture introduced, and any air admitted immediately swept out by means of a rapid current of carbon dioxide. The rate of the current of gas is then reduced to about 30 bubbles per minute, and the tube progressively heated, beginning with the copper oxide. The nitrogen evolved is collected, and after about fifty minutes the combustion will be complete, and the tube, still maintained at a red heat, is swept out for an hour with the current at the rate of about 65 bubbles per minute. The results of four parallel determinations of the carbon and nitrogen in potassium ferrocyanide are given, the mean results being $C = 19·32$ and $N = 22·72$ per cent., as against the theoretical $C = 19·49$ and $N = 22·79$ per cent.

C. A. M.

INORGANIC ANALYSIS.

The Action of Aldehyde and Acetone on Mercuric Acetate. A. Lasserre. (*Journ. Pharm. Chim.*, 1905, xxii., 246-249.)—When acetaldehyde is added to an aqueous solution of mercuric acetate, a crystalline deposit of mercurous acetate is produced after some hours. The same result is produced by formic, propionic, and butyric aldehydes, by paraldehyde, acetal, and by sugars with an aldehydic function (arabinose, xylose, dextrose, galactose, mannose, lactose and maltose). On the other hand, no such deposit is obtained with methyl, ethyl, propyl, butyl, or amyl alcohols, with acetone or sugars with an acetonic function (lævulose and sorbose), or with other carbohydrates (saccharose, trehalose, raffinose, glycogen, inulin, dextrin, and soluble starch). The reaction with acetaldehyde takes place both in the hot and in the cold; and the quantity of mercurous acetate formed depends upon the temperature and length of time.

If sodium hydroxide be cautiously added to an aqueous solution of mercuric acetate containing acetaldehyde, and the temperature maintained at about 0° C., a yellowish-red precipitate eventually changing to gray is formed. This substance, when washed and dried on a porous plate in the dark, is an amorphous powder of a faint yellow tint, deflagrating, when heated, to 180° C. in contact with the air. It contains (according to the mean of several determinations) 77·3 per cent. of mercury, approximating to the 76·9 per cent. required by the compound $CH_3.COH.HgO$. and is thus quite distinct from the analogous compound described by Leys (ANALYST, this volume, p. 341).

If acetaldehyde be replaced by acetone, a precipitate of mercuric oxide is formed, but disappears on the addition of an excess of alkali. The liquid, when allowed to stand, yields a white precipitate, which when dried and distilled with dilute phosphoric acid yields acetone, and deflagrates when heated in the air. It contains 81·5 per cent. of mercury, which corresponds with the formula $CH_3.CO.CH_3$, $2HgO$. Ethyl-methyl-ketone yields a yellowish-white powder, whose composition corresponds with the formula $C_2H_5.CO.CH_3$, $3HgO$. C. A. M.

The Separation of Arsenic from Vanadium and Molybdenum. C. Friedheim, O. Decker, and E. Diem. (*Zeit. anal. Chem.*, 1905, xliv., 665-686.)—The following is recommended as the most simple method of analysing a compound containing arsenic, vanadium, and molybdenum, such as, *e.g.*—

$$6(NH_4)_2O,\ As_2O_5,\ 7V_2O_5,\ 11MoO_3+40H_2O.$$

One gram of the substance is treated with 1 to 1½ grams of potassium iodide and 70 c.c. of hydrochloric acid (specific gravity 1·19) in a flask connected with a condenser. The flask is heated with a flame carefully regulated to prevent stoppage of the tube during the distillation of the iodine. Subsequently a larger flame can be used, until after about twenty minutes the whole of the arsenic has passed over. The distillate is first filtered through glass-wool in order to remove the separated iodine, and is then evaporated on the water-bath with the continual addition of chlorine water until it is of the consistency of a syrup, when the arsenic will have been completely oxidized to arsenic acid without loss, whilst iodine chloride is expelled. The residue is dissolved in water, the solution treated with ammonia in excess, and the arsenic precipitated with magnesia mixture.

The residue in the distillation flask contains the molybdenum and vanadium in a reduced state. It is washed into a flask by means of hot water and treated with hydrogen sulphide, which precipitates the molybdenum as trisulphide. This is filtered off, converted into trioxide, and weighed. The filtrate is freed from hydrochloric acid by evaporation, the residue taken up with sulphuric acid, re-oxidized with potassium permanganate, then reduced by means of sulphurous acid, and titrated with potassium permanganate. C. A. M.

A New Reagent for Nickel. L. Tschugaeff. (*Berichte*, 1905, xxxviii., 2520-2521.)—Hitherto the most sensitive reaction for nickel has been the brown coloration obtained with alkali thiocarbonates, but this has the drawback of being considerably influenced by cobalt. The author finds that α-dimethyl glyoxime, $CH_3.C(:N.OH).C(:N.OH).CH_3$, is a more characteristic and sensitive reagent. The solution under examination is treated with ammonia or sodium acetate solution to remove excess of acid, and then boiled with a little of the powdered dioxime. In the presence of a considerable amount of nickel a scarlet precipitate, $NiD.DH_2$ (DH_2 = dioxime), is produced, but when only traces are present the solution is at first coloured yellow, and the red compound does not separate until the liquid cools. The test is capable of detecting 1 part of nickel in 400,000 parts of water, and is not affected by the presence of ten times the amount of cobalt. The salts of cobalt, however, form brown compounds with the reagent, and when they are present in quantity the following method of detecting small amounts of nickel should be employed: The solution is shaken with ammonia in large excess in order to convert the cobalt salts into ammonium compounds, and is then boiled with an excess of the dioxime. The froth at the sides will be scarlet when considerable quantities of nickel are present, but with traces, such as 0·1 mgm. in 500 mgms. of cobalt, the liquid must be cooled and filtered, and the residue, when washed, will be found to have a pink shade. The dimethyl glyoxime can be obtained as a commercial preparation from Kahlbaum. (*Cf.* this volume, p. 352.) C. A. M.

The Iodometric Determination of Aluminium in Aluminium Chloride and Sulphate. S. E. Moody. (*Amer. Journ. of Science*, 1905, xx., 181-184.)—Stock's gravimetric method, based on the reaction—

$$Al_2(SO_4)_3 + 5KI + KIO_3 + 3H_2O = 2Al(OH)_3 + 3K_2SO_4 + 6I,$$

and ignition of the aluminium precipitate, is shown to give excellent results, and the author has therefore devised the following more rapid volumetric method, in which the liberated iodine is determined: Twenty-five c.c. of the approximately $\frac{N}{10}$ solution of the neutral aluminium salt to be analysed are mixed with 10 c.c. of a neutral solution of potassium iodate (30 grams per litre) and 1 gram of potassium iodide in a Voit's distillation-flask, and the liquid heated for fifteen to twenty-five minutes until nearly colourless, whilst a current of hydrogen is passed through it. The liberated iodine is collected in a Drexel's flask containing water with 3 grams of potassium iodide in solution. Finally, the iodine that has passed over and that left in the Voit's flask is titrated with standard sodium thiosulphate, and calculated into the corresponding amount of alumina in accordance with the equation. The results thus obtained are concordant and in close agreement with those obtained by the gravimetric method. C. A. M.

Estimation of Manganese in Presence of Chromium. Max Gröger. (*Chem. Zeit.*, 1905, xxix., 987.)—The author has tested a modification of Volhard's method for estimating manganese in presence of iron by removal of the iron through zinc hydroxide, and subsequent estimation of the manganous salt by means of potassium permanganate.

It was found that excess of zinc oxide was necessary to precipitate the chromium and that the chromium hydroxide carried down some manganese. The addition of zinc sulphate prevented this, and the author therefore employed basic zinc sulphate in place of hydroxide. A magma of basic zinc sulphate was prepared by dissolving 288 grams of crystallized zinc sulphate in 500 c.c. water, and mixing with a solution of 28 grams caustic soda in 500 c.c. The mixed solution must be well shaken each time before using.

The author performed experiments on (*a*) mixtures of manganese sulphate and chrome alum, (*b*) manganese sulphate and potassium dichromate, (*c*) potassium manganite and potassium dichromate, and (*d*) manganese chromate. In the last three cases the mixture was first reduced in presence of sulphuric acid by sulphurous acid, and the excess of the latter removed by evaporation. Quantities of about 6 grams of the two substances were used in each experiment.

The residues, etc., in the original mixture were dissolved in water and made up to a litre. A certain fraction of this solution was then taken and the zinc sulphate magma added until the precipitate was no longer green but gray-violet in colour, the mixture being kept on a boiling water-bath during precipitation. It was then cooled, made up to 250 c.c., well mixed, and filtered through a dry filter. Fifty c.c. of the filtrate were heated to boiling in a good-sized flask, with a solution of 20 grams of crystallized pure zinc sulphate in 150 c.c. of water, and a standard solution of potassium permanganate (1 c.c. corresponding to 0·001746 grams manganese) was run in, until a permanent rose colour was obtained.

The author gives figures which show that the method is quite reliable as long as, in the portion of solution used, the amount of chromium is not much above 0·1 gram. If the amount is greater, the chromium hydroxide is apt to carry down manganese with it. E. K. H.

The Use of Zinc Hydroxide as a Reagent. Bacovesco. (*Bull. Pharm. Chim. de Roumanie*, 1905, 11; *Ann. de Chim. anal.*, 1905, x., 358, 359.)—Zinc hydroxide mixed with about 5 parts of water gives characteristic reactions with aqueous solutions of different salts. *Mercuric chloride* yields a rose-coloured precipitate turning red after some time, the reaction only taking place in the cold. With *mercuric nitrate* there is a reddish precipitate, and with *mercurous nitrate* a light yellow precipitate, which turns black on boiling. With *copper chlorides* the whole of the copper is precipitated, even in the cold. The soluble salts of *lead*, *cadmium*, *silver*, and *manganese* do not yield any precipitate either in the hot or the cold. *Chromium* salts give a green precipitate of chromium hydroxide, and this is also the case with chromates and bichromates in the presence of sulphur dioxide. With *ferrous* salts the precipitate is white, gradually becoming green; on boiling it changes to yellowish-red. With *ferric* salts there is a blood-red precipitate gradually changing to yellow. *Aluminium* salts give a precipitate. In quantitative analysis, zinc hydroxide can be used to separate copper from cadmium and iron from manganese. C. A. M.

On the Estimation of Sulphur in Pyrites. M. Dennstedt and F. Hassler. (*Zeit. angew. Chem.*, 1905, xviii. 1562.)—In an investigation of Lunge's method of estimation, the authors find that low results are often caused by the formation of basic sulphates of iron during the evaporations with nitric acid; these are insoluble in dilute hydrochloric acid. If, after the second evaporation, Lunge's direction to add

"1 c.c. concentrated hydrochloric acid and 100 c.c. hot water" is strictly followed, these basic sulphates are not completely dissolved.

On the other hand, if the 1 c.c. hydrochloric acid is added first, and the mixture warmed gently for a short time before the 100 c.c. of water are added, any basic sulphates present are easily dissolved.

By this simple means a common and principal cause of a low sulphur result is avoided.

For their analyses during these investigations the authors used the simplified combustion method previously described by one of the present authors (ANALYST, this vol., p. 348). A. N. C.

A New Method for the Estimation of Sulphur in Coal. O. Brunck. (*Zeit. angew. Chem.*, 1905, xviii., 1560.)—This method depends on the combustion of the coal mixed with cobaltic oxide in a current of oxygen.

One gram of the finely-powdered coal is intimately mixed with 2 grams of a mixture of 2 parts cobaltic oxide to 1 part sodium carbonate. The mixture is placed in a small platinum boat and inserted in a combustion tube, open at one end, and fitted at the other with a leading tube, through which the current of oxygen is supplied.

When the current is started, the tube is gently heated with a small flame till the contents of the boat begin to glow. At this point the burner is removed, and the heating only renewed for a short time, when the combustion is almost complete. The whole process is finished in about a quarter of an hour.

The contents of the boat are extracted with warm water and filtered, a few drops of sodium carbonate being added to the wash water to prevent any oxide from passing through the filter.

The filtrate is warmed with a few c.c. of hydrogen peroxide acidified with hydrochloric acid, and the sulphate precipitated in the usual way.

The results are satisfactory, and agree well with those obtained by Eschka's method, or by combustion in a closed bomb.

Care must be taken that the cobaltic oxide used is free from sulphur. It can be prepared pure by gently igniting the dried nitrate. A. N. C.

The Differentiation of Nitrates from other Oxidizing Agents by the Diphenylamine Reaction. C. G. Hinrichs. (*Bull. Soc. Chim.*, 1905, xxxiii., 1002-1005).—If concentrated hydrochloric acid be used in place of sulphuric acid in this test, no blue coloration is given by nitrates until the temperature is raised to 50° C., or to 100° C. in the case of very dilute solutions. Nitrites, on the other hand, are decomposed by concentrated hydrochloric acid at the ordinary temperature, and so give the blue coloration without heat being applied. In order to demonstrate the absence of other oxidizing agents, the solution under examination is mixed with an equal volume of concentrated hydrochloric acid, and then with one drop of a solution of diphenylamine in glacial acetic acid. A blue coloration is produced in the cold by nitrites, peroxides of hydrogen, sodium, barium, manganese, and lead; chromates, bichromates, molybdates, vanadates, and permanganates; chlorates, perchlorates, iodates, ferricyanides, etc. In the case of iodates, the colour has a greenish shade changing to brown. The reaction is very sensitive and rapid, except in the case of barium peroxide, which requires nearly a minute for the development of the blue colour. Should no blue coloration have been obtained in this way, the test is repeated with concentrated sulphuric acid, and an immediate blue coloration then indicates the presence of nitrates.

The reactions of nitrates with brucine and ferrous sulphate are also only produced after heating when concentrated hydrochloric acid is substituted for sulphuric acid. Nitrites do not give any reaction with brucine, but if a little of a vanadate solution be added to the brucine, and several drops of concentrated hydrochloric acid introduced, they give a bright red coloration. Ferricyanides, etc., behave in an analogous manner.

C. A. M.

The Preservation of Solutions of Sodium Hydrosulphite. A. L. Lumière and Seyewetz. (*Bull. Soc. Chim.*, 1905, xxxiii., 931-944.)—Solid sodium hydrosulphite alters very rapidly in moist air, but the anhydrous powder can be kept unchanged in a stoppered flask. In solution it undergoes alteration even in the absence of air, slowly in a 3 per cent. solution, and rapidly in concentrated solutions. The tribasic phosphate of sodium is the most effective agent for retarding the decomposition, and then come sodium silicate, potassium and sodium carbonates, ammonia, and methylamine. Other preservative substances, which, however, cause the hydrosulphite to lose its power of decolorizing indigo in the cold, are trioxymethylene in the presence of sodium sulphite, formaldehyde, acetaldehyde, hexamethylene, tetramine and benzaldehyde. In the presence of these substances the reduction of indigo by the reagent is only effected at a temperature of about 100° C.

C. A. M.

Procedure for Solution and Reprecipitation of Precipitates. F. A. Gooch. (*Zeits. anorg. Chem.*, 1905, xlvi., 208.)—Solution of difficultly soluble precipitates, which have to be collected and washed on a filter, is facilitated by the use of the two simple devices shown. The first, which is used with ordinary funnels, consists of a circular piece of platinum gauze (Fig. 1), cut and bent so as to form a cone having an angle of nearly 60°. This is placed inside the filter-paper in the funnel, the spring of the gauze making it fit the paper very closely. Practically the whole of the precipitate may easily be washed into the platinum cone. To dissolve the precipitate, the cone is lifted by means of ivory-tipped forceps into the vessel in which solution is to be made, any traces of precipitate adhering to the filter-paper being easily washed into the same vessel by means of a jet of water. The second figure shows a perforated platinum plate, which is used in a similar way in connection with Gooch crucibles, the disc being placed above the asbestos in the crucible.

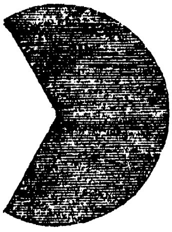


Fig. 1.

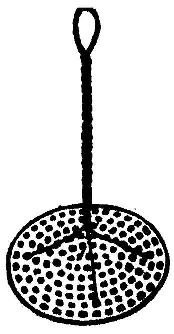
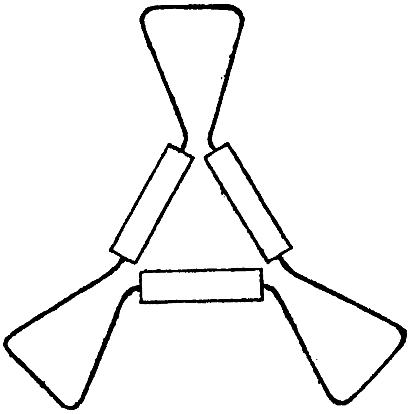


Fig. 2.

A. G. L.

APPARATUS.

A Practical Form of Crucible Triangle. Hermann Lienau. (*Chem. Zeit.*, 1905, xxix., 991).—In the ordinary form of pipe-clay triangle, the clay tubes are only very slightly larger in internal diameter than the wire. Through the expansion of the latter on heating, the pipe-clay is frequently broken up, and the triangle spoilt.



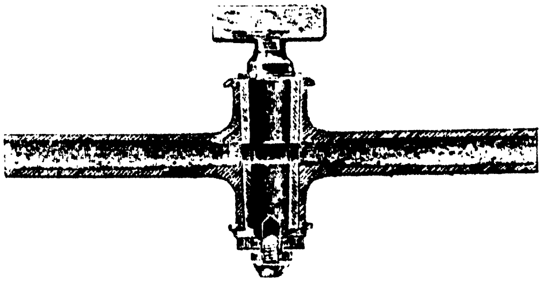
The author recommends triangles of the kind shown in the figure. The chief advantages it possesses are that it can be easily made; the diameter of the wire can be chosen by the analyst; the wire is in a single piece with the joint inside one of the clay tubes; it also has a greater stability on a tripod, or ring-stand, than the ordinary form, as it possesses six points of contact instead of three; and it can be more easily transported with a crucible *in situ*.

E. K. H.

The Obliteration of the Colours in Graduated Glass Instruments. A. Kühn. (*Chem. Zeit.*, 1905, xxix., 990.)—A colour for all kinds of scales which resists the action of boiling water, boiling oil, sulphuric acid at 500° C., and does not require burning in, has been found after long research by the firm of Dr. Kiebert and Kühn, Cassel.

E. K. H.

A Tap for Glass Apparatus whilst using Alkaline Liquids. Lassar-Cohn. (*Chem. Zeit.*, 1905, xxix., 901.)—Ordinary glass taps cannot be used satisfactorily where they come into contact with alkaline liquids, and metal taps cannot be fused on the glass parts.

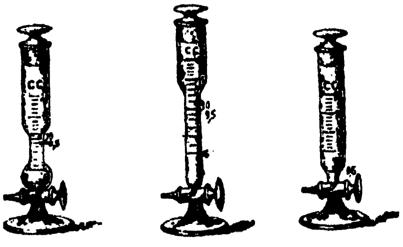


The author tried a modification of a glass tap by employing a hard rubber tap-plug, but that "stuck," owing to the formation of water-glass. A rubber shell was then placed in the glass body in which the hard rubber tap-plug could turn, but it was found impossible to make such a tap tight. The author finally had recourse to phosphor bronze, which is very resistant to alkaline liquids, and replaced the hard rubber parts by this material (see figure).

This tap can be obtained from Dr. R. Muencke, Berlin, N.W.

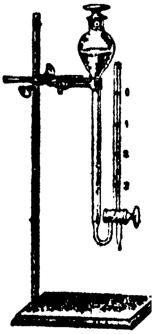
E. K. H.

Testing Apparatus for Butter and Margarine. C. Kippenberger. (*Zeit. angew. Chem.*, 1905, xviii., 1024).—In determining sesame oil in margarine, it is often difficult to separate all the coloured solution obtained by shaking the fat with hydrochloric acid owing to the solidification of the fat.



This apparatus is provided with a stop-cock, through which the whole of the coloured liquid can be drawn off, and is also conveniently graduated for the measurement of the fat and necessary reagents. It is made in three forms, all of which can be placed in the water-bath if necessary. Of the three, the author has found form C the most convenient. A. N. C.

A Burette for the Amyl Alcohol in Gerber's Fat Separator. C. Kippenberger. (*Zeit. angew. Chem.*, 1905, xviii., 1025.)—This is a capillary burette, conveniently graduated, and connected by a double cock with a supply tube.



Only filtered amyl alcohol must be used with it.

The apparatus is made by the firm of C. Gerhardt, of Bonn.

A. N. C.

* * * * *

REVIEW.

CHEMISTRY FOR ENGINEERS AND MANUFACTURERS. By BERTRAM BLOUNT, F.I.C., and A. G. BLOXAM, F.I.C. Vol. II. London: Charles Griffin and Company, Limited. 1905.

This text-book has been thoroughly revised and brought up to date, a good deal of entirely new matter having been included in the second edition recently published.

Among the more important additions will be found descriptions of the Contact Process for the Manufacture of Sulphuric Acid, Electrolytic Processes for Alkali Manufacture, and Improvements in Incandescent Gas Lighting.

As a practical text-book, giving accurate and concise explanations of the principles which underlie the manufacturing operations which are described, it is of the highest value.

The descriptions of the various manufactures dealt with are unburdened by detail, and are remarkably clear.

The book contains a fund of well-compiled and reliable information.

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DECEMBER, 1905. Vol. XXX., No. 357.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE DETERMINATION OF OXYGEN IN COPPER.

By L. Archbutt, F.I.C.

(*Read at the Meeting, November* 1, 1905.)

In June, 1900 (Analyst, vol. xxv., p. 253), I had the honour of submitting to this Society the results of a critical investigation of Hampe's method of determining oxygen in copper. That method consists in heating very fine filings of the clean copper to redness, first in air-free CO_2, and afterwards in hydrogen, carefully weighing the copper before and after each heating. The heating in CO_2 results in a trifling loss of weight, due to the expulsion of, chiefly, superficial moisture, and the heating in hydrogen burns the combined oxygen completely out of the copper, and enables it to be determined by calculation from the loss of weight. I proved by numerous experiments that the process is accurate; that there is no need to use such fine filings as Hampe directs, bright turnings small enough to pass through a tube $\frac{1}{8}$ inch in diameter being fine enough; and that the loss of weight by heating in CO_2 is so trifling that for commercial purposes that part of Hampe's process may be omitted. I was further enabled to show, by the kind assistance of Mr. Blount, that the process of heating the copper to redness in hydrogen and determining the oxygen by the loss of weight gives practically the same results as are obtained by Blount's process of fusing the copper in hydrogen and weighing the water produced in absorption tubes.

Last March, Mr. S. Dickson (Analyst, vol. xxx., p. 145) described a process devised by himself in which the copper is melted in hydrogen with about twice its weight of tin (previously fused in hydrogen). The object of adding the tin is to lower the melting-point of the copper and thus to obtain the advantage, if any, of working on molten copper, without the necessity of having to employ the high temperature necessary to melt copper when unalloyed. I do not propose to criticise Dickson's ingenious process, except to point out that it seems to me to unnecessarily complicate a very simple determination, but I wish to direct attention to an error in his paper which needs correction.

Mr. Dickson stated that unless the copper is very finely divided the whole of the oxygen cannot be expelled from it by heating to redness in hydrogen. By heating copper in thin strips measuring only $\frac{1}{32}$ inch by $\frac{1}{16}$ inch in cross section, he failed to obtain more than 53 per cent. of the total oxygen, and by heating thin copper wire, measuring as little as 0·035 inch in diameter, he obtained only 83 per cent. of the total oxygen. These results were so entirely opposed to my own experience that I felt convinced there must be an error somewhere, but, not having made actual experiments, I was not in a position to contradict Mr. Dickson's assertion. I have now made very careful experiments, and have proved that the thickness of the pieces of copper affects the accuracy of the method very little, if at all; it chiefly influences the time required to carry out a determination.

For the first experiment, I took a piece of copper containing—

Arsenic	0·46 per cent.
Lead	nil.

By heating fine turnings of this copper to redness in hydrogen, I obtained 0·17 per cent. of oxygen. Some of the copper was then sawn into strips, measuring from $\frac{1}{16}$ inch to $\frac{1}{8}$ inch in thickness, and these were heated in hydrogen to bright redness in a silica bulb. In two hours I obtained 0·18 per cent. of oxygen. I then obtained a further quantity of fine turnings and sawings, which I heated to bright redness in a silica bulb, and found 0·18 per cent. of oxygen. Finally, I had some of the copper cut into rough $\frac{1}{4}$-inch cubes and heated in a tube made from Jena glass combustion-tubing. By five hours' heating to bright redness in hydrogen, I obtained 0·17 per cent. of oxygen. The above results are set out in detail in Table I.:

TABLE I.

	Weight of Copper taken (Grms.).	Loss of Weight (Grm.).	Oxygen. Per Cent.
1. *Turnings*	35·242		
1 hour at red heat		0·0581	0·165
2. *Strips, $\frac{1}{16}$ inch to $\frac{1}{8}$ inch thick*	23·9789		
1 hour at bright-red heat (about 820° C.) ...		0·0406	0·169
1 hour more at bright-red heat		0·0420	0·175
1 hour " "		0·0420	0·175
3 hours.			
3. *Turnings and sawings*	35·252		
1 hour at bright-red heat		0·0634	0·179
1 hour more at bright-red heat		0·0640	0·182
1 hour " "		0·0640	0·182
3 hours.			
4. *Rough $\frac{1}{4}$ inch cubes*	30·649		
1 hour at bright-red heat ...		0·0392	0·128
1 hour more at bright-red heat		0·0510	0·166
1 hour " "		0·0517	0·169
2 hours " "		0·0521	0·170
5 hours.			

For the second experiment I took another piece of copper containing—

Arsenic	0·43 per cent.
Lead	0·007 ,,

Some turnings from this copper, heated to redness in hydrogen in a glass bulb, gave 0·12 per cent. of oxygen. I then had a cylinder turned ½ inch in diameter and 1⅝ inches long. The fine turnings of copper produced in making this cylinder were heated in a silica bulb, and gave 0·12 per cent. of oxygen. The solid cylinder itself was then heated to redness in a current of hydrogen in Jena glass combustion-tubing, and after nine hours' heating gave 0·12 per cent. of oxygen. It will be observed that this ½ inch cylinder gave up nearly all its oxygen at a red heat in four hours. By prolonged heating, and increasing the speed of the current of hydrogen to about 3 litres per hour, the remainder of the oxygen was obtained. The results of the experiments with this copper are given in Table II. :

TABLE II.

	Weight of Copper taken (Grms.).	Loss of Weight (Grm.).	Oxygen. Per Cent.
1. *Turnings*	35·305		
1 hour at red heat		0·0436	0·123
2. *Turnings from cylinder*	30·135		
1 hour at dull red heat		0·0356	0·118
1 hour more at dull red heat		0·0356	0·118
1 hour at bright red heat		0·0363	0·120
3 hours.			
3. *Cylinder ½-inch diameter*			
1 hour at red heat		0·0228	0·046
1 hour more at red heat ...		0·0418	0·085
1 hour ,, ,, ...		0·0508	0·103
1 hour ,, ,, ...		0·0528	0·107
2 hours more at bright red heat		0·0518	0·105
2 hours more at low red heat		0·0538	0·109
1 hour more at bright red heat		0·0578	0·117
1 ,, ,, ,, ,,		0·0575	0·116
10 hours.			

For a third experiment I took a piece of copper containing—

Arsenic...	0·49 per cent.
Lead	nil.

I determined the oxygen in this by Dickson's method, fusing the copper in hydrogen with twice its weight of tin, and weighing the water formed in an absorption tube. In two experiments, I obtained 0·12 per cent. of oxygen. I then had a solid cylinder, ½ inch in diameter, turned from this copper, and heated some of the turnings to a moderate red heat in hydrogen in a silica bulb. In one hour I obtained 0·12 per cent. of oxygen—exactly the same as in Dickson's process. I then heated the solid cylinder for ten hours altogether to a good red heat in hydrogen, and obtained 0·12 per cent. of oxygen. The details of this experiment are stated in Table III.:

TABLE III.

	Weight of Copper taken (Grms.).	Weight of Water formed (Grm.).	Oxygen. Per Cent.
1. *By Dickson's method*	10·0	0·0139	0·123
2. " " "	10·0	0·0136	0·121
		Loss of Weight (Grm.).	
3. *Turnings from cylinder*	35·1956		
1 hour at red heat		0·0428	0·122
4. *Cylinder ½-inch diameter*	39·1182		
4 hours at bright red heat		0·0365	0·094
2 hours more at bright red heat		0·0410	0·105
2 " " " "		0·0450	0·115
2 " " " "		0·0453	0·116
10 hours.			

The oxygen determined by heating the ½-inch diameter cylinders of copper to redness in hydrogen was in both experiments a trifle lower than that obtained by heating the turnings from the cylinder, which might have been partly due to a slight superficial oxidation of the turnings, caused by their becoming heated in the lathe, and also to the fact that the turnings, having a much greater surface than the cylinder, would condense a heavier film of atmospheric moisture. The fact that, in Experiment 3 of Table I., the turnings mixed with fine sawings gave the highest result supports that view. Thus, it is quite possible that the results obtained by heating the copper in large pieces may be more accurate than those obtained by heating the fine turnings and sawings, but, in any case, the difference is so small as to be of no practical importance.

The results of the foregoing experiments prove, I think, conclusively that the oxygen in copper can be determined by simple heating to redness in a current of hydrogen, even if the pieces of copper be ½ inch in thickness, if sufficient time be allowed. I have, however, made further experiments with some copper containing over 0·2 per cent. of oxygen. This copper also contained:

Arsenic...	0·43 per cent.
Lead	nil.

By heating turnings of this copper to redness in hydrogen, I obtained from 0·224 to 0·226 per cent. of oxygen in three separate experiments. I then heated a cylinder of the copper, $\frac{3}{8}$ inch in diameter, in the same manner, and in eight hours I obtained 0·21 per cent. of oxygen. I next had some of the turnings carefully rinsed with ammonia to remove superficial oxide, well washed with water, dried with alcohol and ether, and heated to very dull redness in pure CO_2. The turnings after this treatment were heated to redness in hydrogen for two hours, and gave 0·220 per cent. of oxygen. These experiments are recorded in detail in Table IV.:

TABLE IV.

	Weight of Copper taken (Grms.).	Loss of Weight (Grm.).	Oxygen. Per Cent.
1. *Turnings from cylinder :*			
1 hour at red heat	35·0332	0·0789	0·225
1 ,, ,, ,, ...	35·1104	0·0794	0·226
1 ,, ,, ,, ...	30·1153	0·0677	0·224
2. *Cylinder $\frac{3}{8}$-inch diameter*	19·5470		
2 hours at red heat		0·0255	0·130
2 hours more at red heat		0·0343	0·175
2 hours more at bright red heat		0·0402	0·206
2 ,, ,, ,, ,, ...		0·0410	0·210
2 ,, ,, ,, ,, ...		0·0410	0·210
3. *Turnings from cylinder, after being washed with ammonia and heated in CO_2*	25·059		
1 hour at red heat		0·0551	0·220
1 hour more at red heat		0·0551	0·220

This specimen of copper contained an unusually high percentage of oxygen, yet there is a difference of only about 4½ per cent. between the loss of weight of the cylinder and the loss of weight of the clean, dry turnings, which is only one-quarter as much difference as Dickson found when working with wire 0·035 inch in diameter, although the cylinder was more than ten times greater in diameter than the wire. Whether this small difference is really due to a small quantity of residual oxygen left in the copper, I cannot say. On breaking the cylinder in the vice, the fracture presented the same appearance from the core to the outside edge. A photograph of the fractured surface, × 7, is shown in Fig. 1.

Copper which has been deoxidized by heating in hydrogen becomes very brittle; it also expands considerably a $\frac{1}{2}$ inch diameter cylinder expanding as much as $\frac{1}{40}$ inch, and the outer surface is frequently seen to be fissured by cracks. Microscopic examination of polished sections shows that these cracks extend for some distance into the interior of the copper, and pass between the crystalline grains. These grains increase in size as the result of the prolonged annealing, and the junctions between them become loosened. It is, therefore, not surprising that the copper becomes very brittle; nor is it at all surprising, I think, that the oxygen gradually becomes burnt out from the interior of a thick piece. Fig. 2 illustrates the result of bending the bar of copper used in the experiments given in Table IV., before and after annealing in hydrogen.

For microscopic examination, transverse sections were cut from the cylinder used in the experiments detailed in Table III., before and after annealing in hydrogen. The sections were carefully polished, and were etched by immersion in dilute ammonia, to which a few drops of hydrogen peroxide were added.

Fig. 3 shows a section of the deoxidized copper × 6. The cracks proceeding inwards from the outer surface are plainly seen. Fig. 4 is a portion of this section × 50, taken near the edge, and shows the cracks more highly magnified.

Fig. 5. shows the original copper, and Fig. 6 the deoxidized copper, × 50. The enlargement of the crystals and the dark meshing in the deoxidized copper can be plainly seen. Figs. 7 and 8 (× 150) and Figs. 9 and 10 (× 350) show the same specimens under higher magnification.

I also exhibit some high-conductivity copper wire kindly sent me by Mr. A. E. Lewis, of Birmingham. This wire originally contained 99·90 per cent. of copper, and 0·02 per cent. of oxygen; it was quite soft, and could be bent or twisted in any direction and doubled up without breaking or cracking. The specimen exhibited has been deoxidized by heating to redness in hydrogen, and has become so brittle that it can scarcely be bent at all without breaking.

I wish to thank my assistants for their help in making the experiments recorded in this paper.

DISCUSSION.

The PRESIDENT (Mr. Bevan) asked what amount of difference in the percentage of oxygen might be taken as having a marked effect on the physical properties of the copper. Mr. Archbutt had mentioned the great alteration which took place when a sample containing 0·02 per cent. of oxygen had its oxygen removed; would there be a similar difference between a sample containing 0·16 per cent. and one containing 0·14 per cent.? Apart from speed and convenience, the value of a given method would depend upon its accuracy, and if 0·14 and 0·16 were for practical commercial purposes the same, the process which took the shortest time would be the most advantageous. The experiments on the sawings and turnings certainly seemed to show that a good deal of the oxygen was contained on the surface, and in that case, if great accuracy were required, even a somewhat lengthy process might have many advantages.

Mr. DIBDIN said that the differences which had been observed with regard to brittleness showed that this investigation was of far-reaching importance, apart from the question of the analytical method of determining the oxygen. The application of photo-micrographic methods promised important results in the future.

Mr. J. H. B. JENKINS remarked that some years ago the amount of oxygen present in a copper was judged from the specific gravity of the copper. No doubt there was some correspondence between the specific gravity and the proportion of oxygen in copper, but it was a very crude method. Then came the method of digesting the copper in a neutral solution of argentic nitrate, which was generally adopted for the

PLATE I.

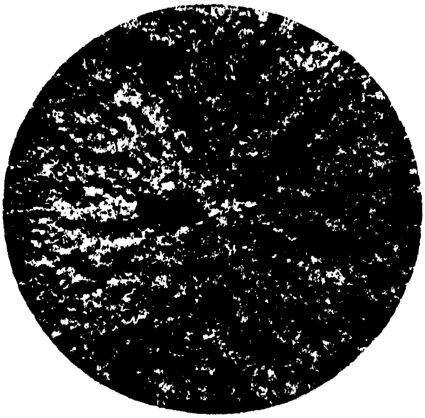


FIG. 1.—FRACTURE OF DEOXIDIZED COPPER BAR (FIG. 2). ×7.

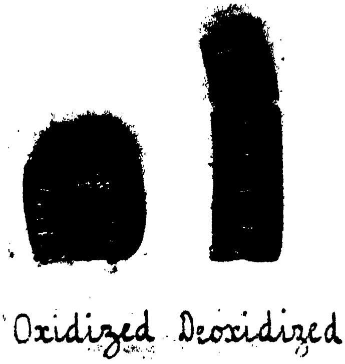


FIG. 2.—RESULT OF BENDING COPPER BARS.

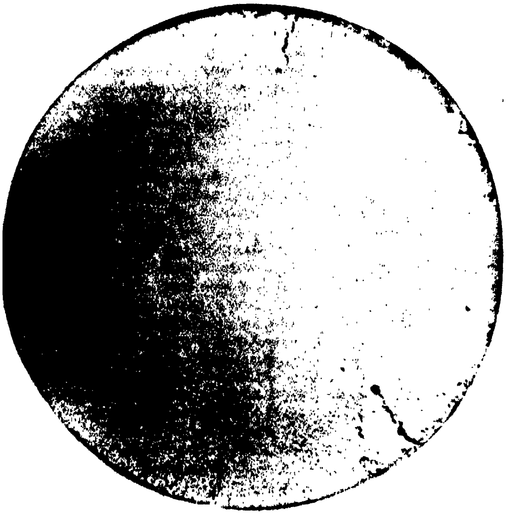


FIG. 3.—DEOXIDIZED COPPER. ×6.

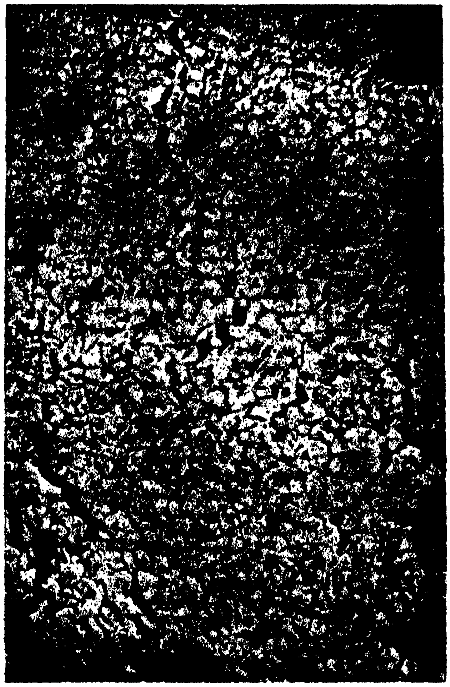


FIG. 4.—PART OF FIG. 3. ×50.

PLATE II.

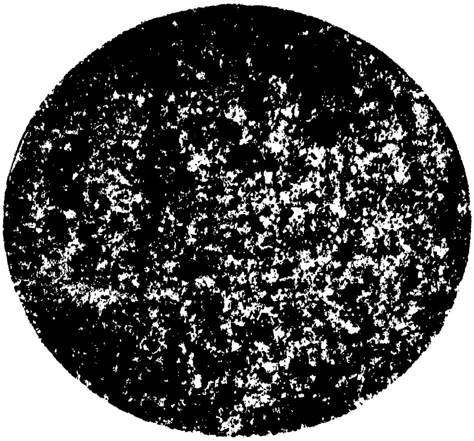


FIG. 5.—ORIGINAL COPPER. × 50.

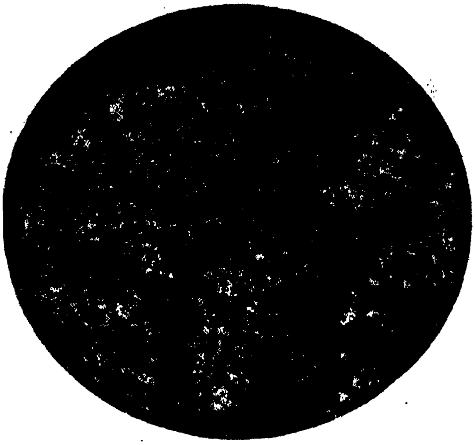


FIG. 6.—DEOXIDIZED COPPER. × 50.

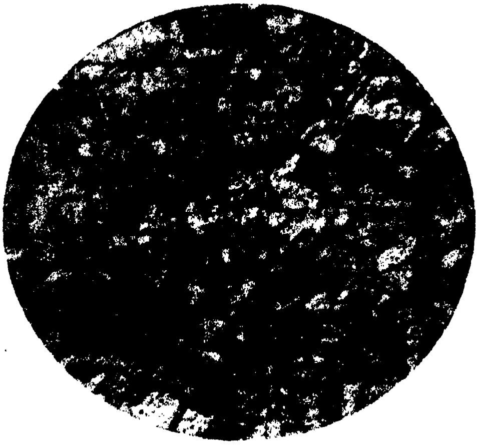


FIG. 7.—ORIGINAL COPPER. × 150.

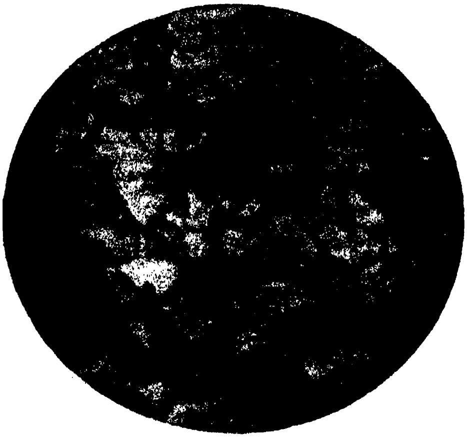


FIG. 8.—DEOXIDIZED COPPER. × 150.

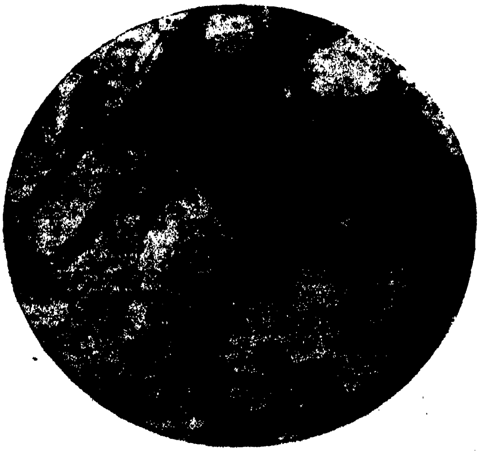


FIG. 9.—ORIGINAL COPPER. × 350.

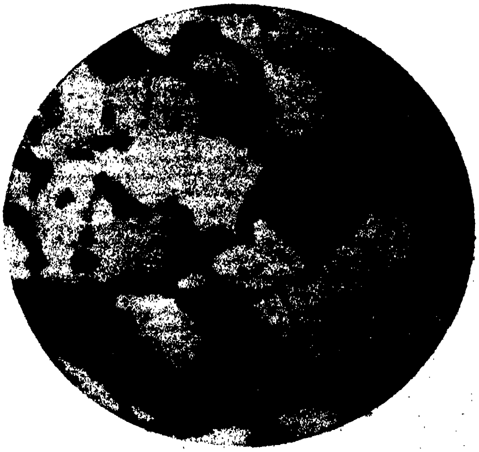


FIG. 10.—DEOXIDIZED COPPER. × 350.

want of anything better. Next came Hampe's method of ignition in hydrogen, which was familiarized to us by the two modifications made by Mr. Blount and Mr. Archbutt respectively. The difficulty experienced in carrying out Mr. Blount's method lay in the high temperature necessary to melt the copper, an injector furnace and porcelain tube being used. This difficulty had been more recently overcome by Mr. Blount in his admirable electric furnace; and now Mr. Dickson had shown a way of obviating the difficulty by reducing the melting temperature of the copper by addition of tin. He had been surprised at Mr. Dickson's statement, that he failed to get the whole of the oxygen in copper wire by Mr. Archbutt's method, because, in his experience, the action of the hydrogen on copper at a red heat seemed by no means so restricted, though thick pieces required more time before the whole of the oxygen was removed. He did not know if Mr. Archbutt agreed with him in thinking that the cuprous oxide, in the quantities in which it was present in ordinary commercial copper, did not reside entirely round the contours of the grains, but that some of it was actually embedded in the grains themselves. One had to conceive that somehow or other the hydrogen found its way, not only along the grain junctions, but also between the foliations of the crystals. This seemed to be indicated in one of Mr. Archbutt's slides, in which the black markings were not confined to the contours of the grains. He thought that so small a proportion as 0·02 per cent. of oxygen could scarcely have any effect on the copper, but that the difference in the case Mr. Archbutt had referred to must be due entirely to the breaking up of the structure of the copper by the action of the hydrogen at a high temperature. Electrolytic copper could be obtained with practically no oxygen, and apparently, with such pure copper, the presence of oxygen was unnecessary. It was only when certain other impurities were present that the oxygen played its part.

Mr. Fairley thought that it might be worth while to try the effect of heating the copper in a neutral gas. He did not know whether nitrogen was a neutral gas to copper at a red heat; nitrides were formed with certain other elements. Was it possible that hydrogen was in an infinitesimal degree soluble in copper, and that there was a tendency, at all events, to form copper hydride (which would decompose almost as quickly as it was formed), and that these changes would tend to disintegration and weakening of the metal?

Mr. Dickson congratulated Mr. Archbutt on the results he had obtained. It might be pointed out that Mr. Archbutt had determined the loss in the weight of the copper and had assumed that to represent the oxygen. The coppers dealt with, however, contained a certain amount of arsenic, and, although he (Mr. Dickson) did not believe that the arsenic was volatile, it was right to mention that his own work had been done with pure electrolytic coppers containing practically no arsenic. With regard to the effect of the oxygen on the copper, he had known a plate containing 0·22 per cent. of oxygen to fail and crack on rolling, and it was considered in that case that the proportion of oxygen was too high. The oxide of copper appeared on the polished specimen of metal as little blue spots on the red background of copper. A photograph of that produced a black and white picture, the black indicating the oxygen. No etching was necessary; in fact, if copper containing arsenic were etched, the arsenic produced marks even more defined than those due to oxygen. He had made some experiments on the annealing of copper, and had obtained a considerable growth of crystals—not in hydrogen, but in a non-oxidizing atmosphere. Some of the crystals were very large, one growing to the extent of the whole field. The metal in that case, however, was not brittle, so he thought the brittleness must be ascribed to the action of the hydrogen. That hydrogen, to a certain extent, was dissolved in or assimilated by the copper was shown in the fusion method, where, the moment the mass became liquid, a great deal of bubbling occurred, and on cooling there was more bubbling, which, he thought, showed that hydrogen had been absorbed by the liquid metal and was given off on cooling. That, he thought, might account for the brittleness after heating short of fusion for a prolonged period. Of course, the growth of the crystals and the opening up of the metal might allow of the determination of the oxygen by prolonged heating, but his efforts had been directed to devising a method which should be capable of being carried out in the ordinary course of laboratory work, without requiring too high a temperature or a great expenditure of time.

The PRESIDENT asked if Mr. Levy (Mr. Blount being absent) could say what was the condition, with regard to brittleness, of the copper remaining after fusion according to Mr. Blount's method.

Mr. LEVY said that, after fusion, a piece of copper of the size ordinarily used could be bent to a considerable extent without cracking, though he could not say whether it would stand bending right over as Mr. Archbutt had shown. The fused copper was so soft that it could be bent between the figures, which was impossible with the unfused piece. With regard to arsenic, a little seemed to be volatilized in the fusion method, a black deposit appearing in the exit-tube after a time.

Mr. JENKINS said that the fact that the copper was not brittle after fusion seemed to suggest an action similar to that which occurred in steel which was in the condition described as "burnt." "Burnt" steel could not be restored by any process short of fusion. Mr. Blount's method really involved fusion, whereas cooling down the copper from a high temperature, short of fusion, would seem likely to leave it in a "burnt" condition. It did not follow that the brittleness was due to want of oxygen.

Mr. DICKSON said that copper, after fusion in hydrogen, was not brittle, but could be hammered quite flat without showing any signs of cracking.

MR. ARCHBUTT said that Mr. Dickson's last remark seemed to answer to some extent the question raised by Mr. Fairley, for if the copper were so malleable after fusion in hydrogen, it could hardly be the action of the hydrogen itself that caused the brittleness. The effect of the presence of oxygen in ordinary refined copper was to neutralize the injurious effect of certain other impurities, and a certain amount of oxygen was necessary in order to make the copper tough. A little more than was actually needed for that purpose would do no harm, and a difference of 0·05 per cent., in the region of 0·15 per cent., would be of no moment. But if the proportion of oxygen were more than 0·2 per cent., he was not so sure whether the extra amount of oxygen might not become detrimental. However, a method which, even with large pieces of copper, was accurate within 5 per cent. of the total oxygen present was amply sufficient for all commercial purposes. He had not brought these experiments forward for the purpose of recommending that large pieces of copper should be used, but simply because he had found that Mr. Dickson's statement had been copied into other journals and disseminated abroad, that the determination of oxygen in copper by Hampe's method was not accurate unless the copper was very finely divided—a statement which, as he had shown, was erroneous. He did not wish to blame Mr. Dickson, who had certainly erred in very good company, for Sir Frederick Abel had tried for a long time to estimate the oxygen by heating in hydrogen, but had failed, though he (Mr. Archbutt) could never understand why. Sir Frederick Abel's attempts were recorded in the *Journal* of the Chemical Society. He (Mr. Archbutt) thought that it would be interesting to take the specific gravity of the copper after heating, but finding that it was so full of cracks, he had measured the increase in diameter instead. As to where the oxygen resided in rolled copper he was not at all clear. He did know that in cast copper the oxide formed a meshing round the grains. Ordinary commercial oxidized copper which had been heated and worked did not present this appearance at all, and it looked as though the cuprous oxide was actually absorbed by the copper, these masses of oxide being found in the crystals, and becoming exposed when the section was polished. Oxidized copper always showed these pits, and therefore it seemed fair to connect them with the oxygen. The pits were shown whether arsenic was present or not. He questioned whether it was the removal of the 0·02 per cent. of oxygen alone that made the copper-wire brittle, but he hoped to make further experiments on the effect of heating oxygen-free copper in hydrogen under the same conditions. Heating in a neutral gas, as suggested by Mr. Fairley, would really be annealing; and annealing, so long as the temperature was not too high, would tend to make the copper more malleable and ductile. In the ½-inch bars the fine cracks in the outer surface could be clearly seen, and it was that which had led him to measure the copper; but in one of these bars he fully believed that, when the copper was put away after the experiment, there were no cracks in it—at any rate, they were not discovered until later. In these experiments he had simply used the copper that was available, and, as Mr. Dickson would know, there was not a great deal of copper used in machine construction that was not arsenical. He did not, however, think that this was of very much importance, though no doubt one ought to experiment with various kinds of copper to make such an investigation complete. As to the volatilization of arsenic, some was undoubtedly distilled out of the copper in the form of arsenious oxide, but that became reduced by the hydrogen, and the arsenic condensed in the cool end of the tube and was weighed with the copper.

Mr. Fairley said that the melting-point of copper would probably be a very different temperature from that at which Mr. Archbutt's experiments were carried out, and the action of the hydrogen would probably be different at a red heat from what it would be at a white heat.

Mr. Archbutt said that the copper which had been melted in hydrogen was allowed to cool in hydrogen, so that it did pass through the stage of being heated to a red heat in hydrogen. On the question of the cause of the brittleness he did not feel able to express a definite opinion. He agreed that it offered a field for future work.

Mr. Dickson inquired to what temperature the copper was heated in Mr. Archbutt's experiments.

Mr. Archbutt said that he did not measure it with the pyrometer, but should think about 750° or 800° C. (since measured and found to range from about 720° to 820° C.).

Mr. Dickson said that under certain circumstances the temperature might come very close to the melting-point.

Mr. Archbutt said that, at any rate, the particles of copper did not fuse together.

The President suggested that possibly the difference between Mr. Archbutt's and Mr. Dickson's results were due to difference of temperature. He should have thought, however, that if Mr. Archbutt used a higher temperature than Mr. Dickson he ought to have obtained less oxygen.

Mr. Dickson: No.

The President said that with a higher temperature the copper would be made less porous to the hydrogen, and yet not sufficiently liquid to come within the conditions of Mr. Blount's process.

Mr. Archbutt said that, provided the copper was brought to a red heat, he did not think the actual temperature made much difference. In his own experiments the copper was sometimes heated to a red heat and sometimes to as high a temperature as could be obtained, but there was very little difference in the results.

Mr. Jenkins thought that it was not necessary to have more than about a dull red heat.

* * * * *

DIKA FAT.

By J. LEWKOWITSCH, Ph.D.

(*Read at the Meeting, November* 1, 1905.)

Dika fat, dika butter, dika oil, oba oil, wild mango oil is the fat obtained from the seed-kernels of various kinds of *Irvingia* (*I. gabonensis*, Aubry-Lecomte; *I. Barteri*, Hooper. *Mangifera gabonensis* is also recorded in the literature of this subject as the tree yielding dika fat, but it appears that *Mangifera gabonensis* is identical with *Irvingia gabonensis*). The trees belonging to the *Irvingia* family are indigenous to the West Coast of Africa, from Sierra Leone to Gaboon. They are termed by the natives "udika," "dika," "dita," and "oba." The fruits are known in Gaboon as "iba." By boiling the kernels with water and skimming off the oily layer the natives obtain the dika butter.

Through the kindness of Professor W. R. Dunstan, I received a considerable quantity of both decorticated nuts and seeds. The seeds were stated to be those of *Irvingia Barteri*, Hooker, from South Nigeria. Unfortunately the nuts were in so advanced a state of mouldiness that it was useless to extract the fat from them. For the same reason it was impossible to ascertain the proportion of seeds to shells in the nuts. The mouldy seeds formed 20 per cent. of the total nuts, but this is, of course, much below what would be actually the case.

The decorticated seeds contained 54·3 per cent. of fat. This fat was in a comparatively fresh state, as the percentage of free fatty acids amounted to 3·35 per cent. only.

The following numbers were found:

FAT.

Specific gravity at 40° C. (water at 40° C. = 1) ...	0·9140
Melting point (capillary tube)	38·9
Solidifying point	29·4 – 27·2
Saponification value	244·5
Iodine value	5·2 per cent.
Reichert-Wollny value	0·42 „
Unsaponifiable matter	0·73 „

FATTY ACIDS.

Mean molecular weight	214
Solidifying point	34·8° C.
Stearic acid	absent.

The iodine value is much lower than the one given by Dieterich—viz., 30·9 to 31·3. Since Dieterich also records a low melting-point—viz., 29°—he evidently did not have genuine dika fat in his hands. The iodine number I found is in good agreement with Oudemans' statement that dika fat consists of laurin and myristin only, to the exclusion of olein. It should, however, be mentioned that the absence of olein was only inferred by him from the fact that an ether-soluble calcium salt could not be obtained. Heckel confirmed Oudemans in that he could not obtain an ether-soluble lead salt; but, of course, this cannot be taken as proof for the absence of oleic acid.

The absence of stearic acid, the low mean molecular weight of the total fatty acids, and the somewhat large quantity of insoluble volatile acids obtained in the Reichert-Wollny test confirm in the main the composition given by Oudemans. We must, however, allow a small proportion of olein. Since Oudemans showed by careful examination that palmitic acid is absent, the fat may be taken as consisting of myristin and laurin and a few per cent. of olein.

DISCUSSION.

Mr. CHAPMAN said that in the latex of several of these gutta trees were found higher alcohols, which appeared to be closely allied to cholesterol, and it would be interesting to know if any such bodies occurred, as seemed possible, in the seeds also. He might call Dr. Lewkowitsch's attention to an alcoholic body having the formula $C_{28}H_{46}O$, which had recently been obtained by Marek from the sap of the *Asclepias Syriaca*, and to which the name "Asclepiol" had been given. It seemed not improbable that the unsaponifiable matter, to which reference had been made, might contain some such substance as this.

Dr. LEWKOWITSCH said that he had not examined the unsaponifiable matter, which might contain more or less considerable quantities of phytosterol.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

***Comparative Experiments upon Chemical Preservatives in Milk. H. C. Sherman, A. W. Hahn, and A. J. Mettler.** (*Journ. Amer. Chem., Soc.*, 1905, xxvii., 1060.)—The authors find that milk kept at 20° to 25° C. without any preservative shows a rapid decrease in the percentage of milk-sugar and increase in acidity during the first three to six days, after which the acid fermentation proceeds much more slowly; neither the destruction of lactose nor the formation of acid is entirely completed after four weeks. The development of acidity in milk is distinctly diminished by the addition of about 0·1 per cent. of hydrogen peroxide, sodium fluoride, sodium salicylate, or a mixture of equal parts of boric acid and borax. When present to this extent, any of these preservatives can be determined, by methods described or referred to in the paper, with a probable error of from 1 to 6 per cent. Hydrogen peroxide, however, decomposes so readily that the amount found gives but little indication of the quantity added. A qualitative change in the fermentation, as shown by the ratio of acid formed to lactose destroyed, is caused by sodium fluoride or salicylate. With these preservatives, especially when present in small quantity, an occasional exposure of the samples to air led to irregular results, the final acidity sometimes exceeding that of samples to which no preservative had been added. The other preservatives examined had no such influence.

A. G. L.

The Detection of Cocoanut Oil in Margarine. Knud Jensen. (*Farmaceutish Tidende*, 1903, p. 385.)—When examining margarine for the amount of butter-fat it contained (the Danish Margarine Law fixes the limit at 15 per cent.) in the presence of cocoanut oil, the Danish Government Laboratory have for some years used a method devised in that laboratory, based on the fact that butter-fat contains chiefly butyric, much less caproic, and mere traces of caprylic acids, whilst cocoanut oil consists chiefly of caprylic and capric, no butyric, and only traces of caproic acids. The method is based on the precipitation of caprylic acid by means of a silver salt, preferably the sulphate, in the following manner: Five grams of the dry margarine fat are saponified as usual, and 110 c.c. distilled off, as in the Reichert-Wollny method. One hundred c.c. are filtered and titrated, the remaining 10 c.c. being filtered into a test-tube, and a few drops of a strong solution of silver nitrate added. In the presence of cocoanut oil the liquid will turn milky, otherwise it will only be opalescent. If cocoanut oil be found, another portion of 5 grams of the margarine is saponified and distilled according to the Reichert-Wollny method. The distillate 110 c.c., is neutralized by sodium hydroxide, rendered slightly acid by a few drops of $\frac{N}{10}$ normal sulphuric acid and 1 gram of silver sulphate (or, in the presence of a large proportion of cocoanut oil, 1½ grams) added, when silver caprylate will be precipitated in the course of a few hours. After filtering, 30 c.c. of diluted sulphuric acid (as used in the Reichert-Wollny method) are added, 110 c.c. distilled off, and 100 c.c. of this titrated as usual. The remaining 10 c.c. are filtered into a test-tube, and silver nitrate added to ascertain if all the caprylic acid has been precipitated. W. P. S.

The Volatile Fatty Acids of Cocoanut Oil and Butter. O. Jensen. (*Zeit. Untersuch. Nahr. Genussm.*, 1905, x., 265-283.)—By means of fractional precipitation of their silver salts, the author has separated the volatile fatty acids of cocoanut oil from one another, and describes their properties and solubilities. The proportion in which these acids occur in the two above-mentioned fats is also given. Capric acid is almost insoluble in cold water, whilst caprylic and caproic acids dissolve in water at a temperature of 15° C. to the extent of 0·079 and 0·872 per cent. respectively. If, however, a little caprylic acid be added to a saturated caproic acid solution, almost the whole of the latter is precipitated and replaced by caprylic acid. Butyric acid has no influence on the solubility of caproic and caprylic acids.

It is shown that cocoanut oil contains no butyric acid and but little caproic acid, the volatile fatty acids consisting principally of capric and caprylic acids. Butter, on the other hand, contains on the average 1·88 per cent. of caproic acid and 3·92 per cent. of butyric acid. The quantities of these two acids in butter, however, varies considerably.

The author further proves that the Reichert-Wollny and Polenske values (ANALYST, 1904, xxix., 154) of a mixture of butter with cocoanut oil do not agree with the values calculated from the known amount of each fat present, but that the Kirschner value (ANALYST, 1905, xxx., 205) agrees closely with the calculated value. It should, however, be mentioned that the latter value is generally proportional to the Reichert-Meissl value, and that the method was originally proposed for the detection of butter and cocoanut oil in margarine. For the detection of cocoanut oil in butter, a determination of the caprylic acid by precipitation of the latter, as its silver salt, from the Reichert-Meissl filtrate, is strongly recommended. W. P. S.

The Quantity of Sodium Salts occurring in Wines. O. Krug. (*Zeit. Untersuch. Nahr. Genussm.*, 1905, x., 417-421.)—Results of analyses of forty-eight samples of well-known samples of red and white wines are given, from which it is seen that the quantity of soda (Na_2O) varies from 0·0004 to 0·0060 per cent. In 80 per cent. of the samples the amount of soda present did not exceed 1 per cent. of the total ash. Red wines appear to contain slightly less soda than white wines, but there is very little difference between the two kinds in this respect. The author considers that the presence of more than 0·01 per cent. of soda in a wine points to sophistication, provided that the amount of chlorine is normal and that the suspicion is borne out by the other analytical data. W. P. S.

Modification of Welmans' Method for Sugar and Fat Estimation in Cocoa Preparations. A. Steinmann. (*Chem. Zeit.*, 1905, xxix., 1074.)—Welmans' well-known method, using water-saturated ether and ether-saturated water as solvents, has the following defects: (1) By rise of temperature ether is driven out of the aqueous solution of ether; (2) ether is expelled from the water solution by sugar dissolving therein; (3) during filtration more ether is lost from the aqueous sugar solution, which becomes therefore more concentrated; and (4) by the above expulsion of ether from the aqueous layer, the ethereal solution of fat is diluted.

The author therefore proposes to use petroleum ether in place of ether, as this is not soluble in water, nor does water dissolve in it. He further takes the specific gravity of cocoa fat as 0·95 instead of 1·0, and that of sugar in solution as 1·6 instead of 1·55. These figures only make very slight differences in the result.

To avoid loss of petroleum ether, it should be measured into the separating-funnel by means of a pipette, the end of which remains in the funnel close to the surface of the liquid whilst delivering. The fat solution in petroleum ether is removed with similar precautions, filtered if necessary, and the filter washed with ether into the tared flask.

The author finds the method accurate and trustworthy. E. K. H.

The Composition of the Essential Oil from the Leaves and Stalks of the Sweet Orange. G. Litterer. (*Bull. Soc. Chim.*, 1905, xxxiii., 1079-1081.)—The specimen of oil examined had a specific gravity of 0·8603 at 15° C.; optical rotation ($l = 100$ mm.) of +56° 46′ at 20° C.; and refractive index, n_D, at 20° C. of 1·472. The citral isolated by shaking the oil with sodium sulphite and sodium bicarbonate solution amounted to 4 per cent., whilst the non-aldehydic part, separated by means of ether, had the following characteristics: Specific gravity at 15° C., 0·8582; optical rotation at 20° C., +57° 34′; and refractive index at 21° C., 1·4725. The oil also contained about 20 per cent. of alcohols, partly in the free state and partly as esters, the latter amounting to 4·1 per cent. Geraniol was identified with certainty, and there was also a small proportion of a substance with properties agreeing with those of dextro-rotatory linalol. When saponified and fractionally distilled under a pressure of 10 mm. 60 per cent. of the oil distilled over between 60° and 84° C., and 10 per cent. between 84° and 88° C. On further fractionation of the terpene portion a large proportion of dextro-rotatory camphene and a small amount of limonene were isolated. The fraction distilling between 84° and 88° C. contained the supposed linalol, whilst the residue from the distillation contained the geraniol. C. A. M.

Testing Wheat-flour for Commercial Purposes. Harry Snyder. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1068.)—The author discusses various methods of examining flours, and concludes that the best results are given by the joint application of chemical and baking tests, as chemical tests alone are not always a safe guide to the bread-making value of the flour, and baking-tests alone furnish no absolute data as to grade. The most important determinations are those of ash, moisture, total nitrogen, gliadin nitrogen, granulation, absorptive capacity, and colour. The first and second grades of patent flour never contain more that 0·48 per cent. of ash; straight grade rarely more than 0·55; whilst the first and second clear grades contain 0·8 and 1·75 per cent. respectively. Moisture should not exceed 13 per cent. Total proteids (N × 6·25) vary from 11·5 to 13 per cent.; the best grades contain 12·5 to 13 per cent.; a higher nitrogen content does not appear to be advantageous from the bread-making point of view. On account of variations in the content and composition of gliadin, the value obtained for this body must be used with caution, but for flour milled from the same wheat the lower grades always contain less gliadin than the best grades. The colour of flour has become less important since chemical bleaching of flour has been introduced. It should be noted that in a general way the same factors impart commercial and nutritive value to flour. A. G. L.

The Hydrolysis of Meat Extracts. K. Micko. (*Zeit. Untersuch. Nahr. Genussm.*, 1905, x., 393-415.)—Meat extracts contain little, if any, peptone, which gives the biuret reaction; but it is quite possible that polypeptones are present; and the greater part of the investigation here recorded deals with this question. The polypeptones are best separated by hydrolysis, and the author has employed the esterification method of E. Fischer (see *Journ. Chem. Soc.*, 1901, ii., 780) for this purpose. His preliminary experiments on the hydrolysis of meat extract as a whole are described at some length, and the various amino-esters obtained are identified.

The meat extract was boiled for many hours with an excess of concentrated hydrochloric acid, the mixture evaporated, filtered from the separated salts, again evaporated, and the residue dissolved in hot absolute alcohol, into which solution hydrochloric acid gas was passed to saturation. The solution was then separated into various fractions by distillation under reduced pressure, as described in Fischer's method. Towards the end of the distillation of the alcoholic solution an oily fraction was obtained, which after fractional distillation was found to consist principally of lactic acid esters, succinic acid esters being also present.

The first fraction of the main distillation contained alanin, besides which the presence of glycocoll, and possibly amino-valerianic acid, was suspected. A not inconsiderable part of the crude amino acids consisted of a thick syrupy mass, whose nature was not at the time investigated.

The second fraction also contained alanin as the principal constituent of the amino acids, which were insoluble or difficultly soluble in alcohol. Glycocoll and leucin were also detected, but amino-valerianic acid, whilst probably present, could not be identified with certainty. This fraction, like the first, yielded a considerable quantity of a thick syrup, of which the constituents were not separated. W. P. S.

Coffees without Caffeine. G. Bertrand. (*Ann. de Chim. anal.*, 1905, x., 401.)—The species *Coffea Humblotiana*, which grows in Madagascar, was found by the author to be absolutely devoid of caffeine (ANALYST, xxvi., 188), and the same characteristic is shown by three other species of coffee—*C. Gallienii*, *C. Bonnieri*, and *C. Mogeneti*—each of which contains the bitter principle *cafamarine*, found in *C. Humblotiana*. It is curious that all these species, and also *C. mauritiana*, which contains but little caffeine, should only occur in Madagascar and the neighbouring islands, and not on the adjacent continent. C. A. M.

Note on the Determination of Caffeine. W. A. Puckner. (*Amer. Journ. Pharm.*, 1905, lxxvii., 488.)—In experiments made to determine the conditions under which caffeine may be dried without loss, it was found that a chloroform solution of caffeine (dried at 95° C.) can be evaporated in an open dish at from 50° to 60° C. without appreciable loss of the alkaloid, and that it can also be dried without loss at 95° C. in a distillation-flask, but not in an open dish. The experiments also confirmed Tasilly's conclusion that caffeine does not become anhydrous even at 110° C. If the alkaloid (dried at 95° C.) be dissolved in water or chloroform, and the solution allowed to evaporate spontaneously, the residue can be dried to the original weight over sulphuric acid. C. A. M.

Note on the Determination of Acetanilide. W. A. Puckner. (*Amer. Journ. Pharm.*, 1905, lxxvii., 488, 489.)—The author's experiments show that acetanilide is quite volatile at comparatively low temperatures. It can be determined by allowing its solution in chloroform or ether to evaporate at the ordinary temperature and drying the residue over sulphuric acid. Or, if the solvent be distilled off and the residue dried at 50° to 60° C., there is only a slight loss of acetanilide, and the weight becomes practically constant after twelve to twenty-four hours. At 95° acetanilide is appreciably volatile, even when contained in a narrow flask. The residue obtained, when the solvent is driven off from an ether or chloroform solution, is not pure acetanilide; hence, if loss by vaporization is avoided, high results are invariably obtained. C. A. M.

The Volumetric Determination of Pyramidon and Antipyrin in Mixtures of the Two Substances. G. Pégurier. (*Ann. de Chim. anal.*, 1905, x., 392, 393.) —A slight modification of the volumetric method already described (ANALYST, XXX., 338) enables a simultaneous determination of pyramidon and antipyrin to be made. It is based upon the mono-acidity of pyramidon towards helianthin, whereas antipyrin is neutral towards that indicator. The two substances are first determined together by titration with picric acid in the manner described. A second determination is then made in which 0·231 gram of the sample is dissolved in 10 c.c. of water, and exactly neutralized with $\frac{N}{10}$ sulphuric or oxalic acid after the addition of 2 drops of helianthin indicator. The liquid is then treated with 40 grams of $\frac{N}{20}$ picric acid solution, and titrated with $\frac{N}{10}$ potassium or sodium hydroxide solution, with phenolphthalein as indicator. A simple calculation then gives the respective quantities of pyramidon and antipyrin in 100 grams of the sample. C. A. M.

ORGANIC ANALYSIS.

A Study of the Hydrogen Peroxide Method of determining Formaldehyde. J. K. Haywood and B. H. Smith. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1183.)—Experiments with the Blank and Finkenbeiner method, and with Fresenius and Grünhut's modification of the same, have led the authors to work out the following process: Three c.c. of the formaldehyde solution under examination (of known specific gravity) are added to a mixture of 50 c.c. of N sodium hydroxide and 50 c.c. of 3 per cent. hydrogen peroxide contained in a 500 c.c. Erlenmeyer flask. A funnel is placed in the neck of the flask, which is then heated, with occasional shaking, for five minutes on the steam-bath. At the end of this time it is cooled to room-temperature, and the excess of alkali titrated with N sulphuric acid, using litmus as indicator. This method gives slightly higher and more consistent values than do the older methods, as there is no chance of decomposition of the formaldehyde by the soda alone, leading to the formation of methyl-alcohol; the soda used is also more dilute, and hence can be more accurately measured. A. G. L.

New Colour Reactions of Aldehydes. A. B. Lyons. (*Amer. Journ. Pharm.*, 1905, lxxvii., 492, 493.)—Formaldehyde with sulphuric acid is used as a reagent for morphine, and conversely morphine and sulphuric acid may be employed as a reagent for formaldehyde, the test being capable of detecting 1 part of the aldehyde in 20,000. By the addition of a trace of a ferric salt to the reagent the sensitiveness of the test is greatly increased, and it is then capable of detecting 1 part in 2,000,000. Other aldehydic bodies give analogous reactions, and sucrose and lactose, in particular, may be identified, though not distinguished, in this way. In the case of sucrose (0·1 to 1 per cent. solutions), the violet-blue coloration, obtained as from formaldehyde, is surmounted by a distinctive yellow border. The test for formaldehyde is only applicable to distillates, and is only of negative value. The reagent cannot be used directly with milk even after coagulation, or for the detection of glucose in urine. C. A. M.

A Fuller Study of the Neutral Sulphite Method for determining Some Aldehydes and Ketones in Essential Oils. S. S. Sadtler. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1321.)—The author has revised some of his previous work on the subject (*Journ. Soc. Chem. Ind.*, xxiii., 1904, 303). He finds that citral reacts slowly with 2 molecules of neutral sulphite; cinnamic aldehyde most energetically with 2; benzaldehyde moderately with 1; vanillin slowly with 1; carvone moderately with 2; pulegone moderately with 2; formaldehyde instantaneously with 2; whilst citronellal, camphor, menthone, and fenchone do not react at all. He believes that proximity of double bonds and of benzene nucleus (if present) favour the reaction, and that increasing molecular weight retards it. The method used consisted in measuring the alkali produced by standard hydrochloric acid, rosolic acid being generally used as indicator. The mixture of sample and sulphite solution was heated in a water-bath if necessary, sometimes after the alkali formed in the cold had been neutralized. Kerosene was used as diluent if a large percentage of aldehyde or ketone was present in the sample. A. G. L.

Discovery of a New Terpene Alcohol in Ethereal Oil of Myrtle. H. von Soden and F. Elze. (*Chem. Zeit.*, 1905, xxix., 1031.)—In the manufacture of the commercial substance "myrtol" from Spanish myrtle oil a higher boiling constituent is obtained as a by-product. This consists chiefly of the acetic ester of a new terpene alcohol, $C_{10}H_{18}O$, which the authors call "myrtenol."

To obtain pure myrtenol, the high-boiling by-product is first fractionated under reduced pressure, and the middle fraction, consisting of about 60 per cent. of the whole, is retained. It has a specific gravity of 0·975 at 15° C., an optical rotation of about +28° in a 100 mm. tube, and contains about 80 per cent. of ester reckoned as $CH_3COOC_{10}H_{17}$.

The middle fraction is then saponified with alcoholic potash (1 in 3), the oil obtained distilled with steam, and then rectified under reduced pressure. The crude myrtenol is now converted into the acid phthalic ester by heating for two hours with equal parts of phthalic anhydride and benzene, purified by dissolving in dilute soda solution, and extracting the impurities from the solution with ether. It is then reprecipitated by dilute sulphuric acid, and obtained in a crystalline form, though it is still contaminated with the phthalic ester of geraniol. To remove this it is washed with small quantities of petroleum ether with the aid of the filter-pump, and finally purified by repeated crystallization from ligroin (B.P., 100° to 125° C.). The hard white crystals thus obtained melt at 116° C. To obtain the alcohol, this ester was saponified with potash, the alcohol driven off with steam, and fractionated under reduced pressure. Thus prepared, it boils under 751 mm. pressure at 220° to 221° C., and under pressure at 79·5° to 80° C.; its specific gravity at 15° C. = 0·985; optical rotation in 1 decim. tube = +49°·25′. It is quantitatively esterized by acetic anhydride. It must be considered a primary cyclic terpene alcohol containing one double carbon linking. E. K. H.

A New Reaction for the Detection of Acetone in Urine. V. Frommer. (*Biochem. Centralbl.*, 1905, iv., 357.)—If 10 c.c. of urine be treated with 1 gram of potassium hydroxide, and the alkaline solution mixed with 10 to 12 drops of salicylic aldehyde and heated, a purple-red coloration is produced when acetone is present, whereas otherwise the urine remains unaltered.

C. A. M.

A Characteristic Colour Reaction of Methyl-diamino-triazine. A. Ostrogovich. (*Bull. Soc. de Sciinte, Bucarest*, 1905, xiv., 317-319.)—A trace of this base dissolved in a few drops of alcohol, and mixed with 1 drop of an alcoholic solution of iodine, gives an intense violet or blue coloration when the solvent is evaporated in a current of air at the ordinary temperature. This reaction is only obtained with the free base, whilst basic salts give a more reddish and less pronounced coloration. No reaction is produced with neutral salts alone or in the presence of an acid, or of sodium or potassium hydroxide in excess of that required to liberate the base. Derivatives of methyl-diamino-triazine and other triazines or their derivatives do not give any coloration in the test. C. A. M.

A New Test for Lactic Acid. Croner and Cronheim. (*Biochem. Centralbl.*, 1905, iv., 357.)—This is based upon the conversion of lactic acid into iodoform under the influence of iodine and an alkali, and the formation of isonitrile from the iodoform by the action of a primary amino base. The odour of the final product is so characteristic that very faint traces of lactic acid can be detected in gastric juice in this way without previous isolation by extraction with ether. The use of aniline is recommended in place of methylamine in the final reaction. Obviously alcohol and acetone, which react in the same way as lactic acid, must be eliminated by heating the liquid before the test is applied. C. A. M.

On the Gelatinization of Tung Oil. M. Kitt. (*Chem. Rev. Fett u. Harz Ind.*, 1905, xii., 243.)—In experiments to determine the chemical changes that take place in the gelatinization of tung oil when heated, it was found that there was a loss in weight, which in one case amounted to 8·83 per cent. of the original amount. Part of the gelatinous product was saponified, and the separated fatty acids titrated with alcoholic potassium hydroxide solution, alkali blue being used as indicator. They had an acid value of 185·2, whilst the saponification value, which could not be exactly determined owing to the dark colour, was somewhat above 200. When these fatty acids were heated with a mixture of alcohol and ether a portion remained insoluble, and on treating became agglomerated into a mass, which was also insoluble in benzene, though it dissolved in hot glacial acetic acid. The author's conclusion was that inner anhydrides were formed in the gelatinizing process, and that the loss in weight was due to partial decomposition of the fat and loss of glycerin.

C. A. M.

Copal Oils. L. Schmoelling. (*Chem. Zeit.*, 1905, xxix., 955.)—The author has undertaken investigations of those commercial copal oils which are obtained by the distillation of the two resins most used in the manufacture of lac—viz., Kauri and Manila. As shown by Tschirch, the volatile constituents of these two copals differ considerably, consequently the two oils differ much in composition. The Kauri oil is a mobile liquid of clear yellow colour possessing a pleasant aromatic smell. Its specific gravity is 0·8677 at 15° C. It remains unaltered even after standing for weeks in an open vessel. The Manila oil has a rose colour when freshly prepared, which changes in a few hours to a cherry-red. Its specific gravity at 15° C. = 0·9069.

The two oils are similarly soluble in ether, carbon tetrachloride, carbon disulphide, benzol, amyl alcohol, and turpentine; but the Kauri oil is somewhat less soluble in alcohol, and the Manila oil gives a precipitate with petroleum ether, whilst Kauri oil is completely insoluble in that solvent.

The author gives the following numbers for the two oils:

	Kauri Oil.	Manila Oil.
Acid value	3·0	28·3
Saponification number (cold)	4·9	45·7
Ether number	1·9	17·4
Iodine number (Waller)	288·9	230·4

The acid value and saponification number for Manila oil fall after keeping the oil exposed for some days at the ordinary temperature, whilst the ether number remains constant. This points to volatile acids in the oil.

The oils were then submitted to fractionatal distillation, and the amounts of the fractions were found to be different in the two cases for the same ranges of temperature. In the case of the Kauri oil, practically the whole came over between 150 and 160, whereas the Manila oil gave the following fractions: (1) 68° to 135°, 6 per cent.; (2) 135° to 160°, 16 per cent.; (3) 160° to 185°, 30 per cent.; (4) 185° to 250°, 10 per cent.; (5) over 250°, 34 per cent. No substance resembling pinine or limonene could be detected.

The oils were then exactly neutralized with $\frac{N}{2}$ potassium hydroxide, and driven over with dry steam. The Kauri oil is almost completely volatile with steam. The oil separating in the distillate was removed, and allowed to stand in a warm place for several days to dry. A clear ethereal oil was thus obtained, with an odour closely resembling that of the crude oil. The specific gravity = 0·8633 at 15° C. Of the Manila oil, only about half was volatile with steam, the volatile portion, had a clear yellow colour, and an odour less powerful than that of the crude oil. The specific gravity = 0·8567 at 15° C. The iodine numbers for these distilled oils are: Kauri oil, 307·6; Manila oil, 282·0.

The steam distilled oils were fractionated, and gave the following figures:

Kauri Oil.			Manila Oil.		
1.	152° to 155° ...	21 per cent.	1.	up to 133° ...	26 per cent.
2.	155° to 157° ...	38·5 ,,	2.	155° to 165° ...	26 ,,
3.	157° to 170° ...	24·7 ,,	3.	165° to 170° ...	12·3 per cent. (yellow)
4.	over 170° ...	15·8 ,,			

Two parts of fraction 4 of the Kauri oil, with 8 parts of alcohol and 8 parts of ether, gave, on addition of 1·5 parts of bromine, well-defined crystals after some hours—an indication of the presence of a limonene-like body.

Some observations were made on the aqueous liquid obtained in the dry distillation. In both cases this had a high colour. The acid values of this liquid were for Kauri = 23·8; for Manila, 173·1. The Manila liquid gave reactions for acetic and formic acids, but the presence of these two acids in Kauri liquid could not be detected. The investigation is being continued, and a further communication is promised.

E. K. H.

Recognition of "Blown" Fatty Oils in Mixtures with Mineral Oils.

J. Marcusson. (*Chem. Zeit. Rep.*, 1905, xxix., 278.)—To detect a "blown" fatty oil (*i.e.*, one which has been thickened by action of air at a high temperature—*e.g.*, rape oil, etc.) in a mineral oil the first thing is to ascertain the solubility or insolubility of the separated fatty acids in petroleum spirit. The determination of the Reichert-Meissl value affords another indication, due to the volatile acids formed during oxidation. Conclusions as to the presence of a thickened fatty oil can also be drawn from a determination of the viscosity of the sample, and comparison with Spitz and Hönig's value for the viscosity of pure mineral oil. The iodine value, and the molecular weight of the fatty acids, and the colour reactions of Halphen and Milliau serve to detect blown rape oil or cotton-seed oil, which are the oils almost exclusively used and adulterate oils for lubricating purposes.

To distinguish these, the odour of the sample and of the fatty acids, the consistency of the fatty acids and the behaviour towards ether of the lead-soap obtained from the fatty acids may be used. E. K. H.

Observations on the Elementary Analysis of Crude Petroleum Oils. A. Ostrogovich. (*Bull. Soc. de Sciinte, Bucarest*, 1905, xiv., 314-316.)—The ordinary method of weighing the liquid in bulbs of fusible glass has the drawback that at the close of the combustion particles of carbon may become fused within the glass. To obviate this the author has made bulbs of hard Jena glass of the shape and size shown in Figs. 1 and 2. For light petroleum oils the shape shown in Fig. 1 is used, there being but little loss by volatilization through the long neck during the weighing. The weighed bulb is placed in a platinum boat, and is introduced into the combustion tube with its neck pointing towards the absorption vessels, this facilitating the combustion of the petroleum. The author has found a boat of the form shown in Fig. 3 very serviceable for the combustion of thick petroleum or distillation residues. It is made of hard Bohemian glass tube, the ends being fused together and one bent into a hook. The side is pierced so as to form an opening about 4 mm. wide extending nearly the length of the tube, whilst a flat glass cover is put over the opening during the weighing. In order to prevent the boat turning over in the combustion tube it is placed in a strip of platinum foil, bent twice at right angles, the upright sides coinciding with the height of the tube. C. A. M.

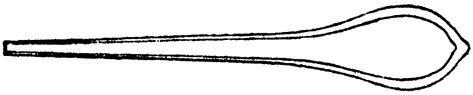


FIG. 1.

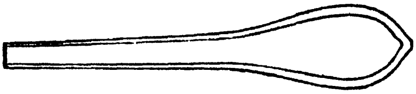


FIG. 2.

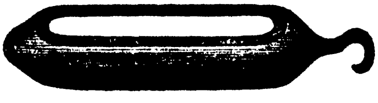


FIG. 3.

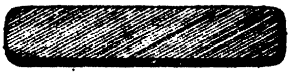


FIG. 4.

Estimation of Oil in Water from Condensing Engines. J. McFarlane and J. Mears. (*Chem. News*, 1905, xcii., 108.)—One or two litres of the water, according as to whether the quantity of oil is above or below 0·4 grain per gallon, are heated nearly to boiling with 5 c.c. of a ferric chloride solution, made by dissolving 10 grams of iron in 200 c.c. of hydrochloric acid, oxidizing with nitric acid, and making up to 1 litre. An excess of ammonia is added, and the whole boiled for two minutes. The precipitate, which contains the whole of the oil, is then filtered off on a fat-free 15 cm. filter-paper, washed with hot water, dried at 100° C., and extracted with ether, the oil being weighed as usual. A. G. L.

Valuation of Tannic Acid for Dyeing and Calico Printing. W. S. Williams. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 877.)—After briefly describing the chief sources of tannin suitable for the purpose, the author discusses their valuation by chemical analysis and practical dyeing tests. For the accurate determination of moisture the sample must be dried *in vacuo* over sulphuric acid, but the results obtained by drying the sample in a layer of not more than 7 mm. deep for two to two and a half hours at 95° C. will be sufficiently correct for most purposes. The dried material should not be used for the subsequent analysis. Resinous or tarry matter may be tested for by boiling with an equal weight of acetic acid, when, if present, a thick, sticky mass will collect on cooling at the top of the solution. After numerous trials with other methods, Hunt's modified permanganate process is considered the most satisfactory for the determination of tannin, the permanganate solution being perferably standardized with pure gallic or tannic acid. For a quick comparison of values the dyeing test is more convenient, and an improved method of applying this test is described, in which a printing paste prepared from the tannin sample is printed on bleached cotton cloth and dried. The paste consists of ½ ounce of the sample dissolved in 1 ounce acetic acid and 3 ounces water, this solution being added to 12 ounces of thickening, made by boiling 4 to 6 pounds light British gum in 1 gallon of water. All the patches to be compared are steamed and fixed by working fifteen minutes at 50° C. in a bath containing 1 ounce tartar emetic per gallon, after which the swatches are rinsed two minutes in cold and two minutes in hot water and dried, leaving only antimony tannate and any colouring matter present in the tannin. A part of each swatch is kept for future comparison, the remainder being dyed in one bath with an excess of a red shade of methylene blue, using the ordinary assistants. When the dyeing is completed, the swatches are removed from the bath, rinsed in cold water, then in water at 90° C. for five minutes, soaped lightly, rinsed, and finally dried. The printed portions of the cloth so dyed give a very fair idea of the mordanting value and clearness of tone to be obtained in practice, the results agree perfectly with those found by analysis. By substituting gallic acid for tannic acid in a test trial no trace of the basic dye-stuff is fixed on the fibre.

W. H. S.

Balata Gum. A. Tscirsch and E. Schereschewski. (*Archiv. Pharm. Vol.*, 243, p. 358; through *Pharm. Journ.*, 1905, Vol. 75, p. 481.)—Balata gum is the dried latex (granular fluid contained in the lactiferous vessels) of *Mimusops globosa*, a tree growing in British and Dutch Guiana. It is at present one of the most important substitutes for gutta-percha, which it strongly resembles in its general properties. The balata examined by the authors yielded to boiling water 1·72 per cent., to boiling alcohol 41·5 per cent., and to chloroform (after alcohol) 45·3 per cent. The water-soluble substances consisted of albumin and a little gum. By treatment with alcohol of different strengths the alcohol-soluble substances were separated into an oily fraction, a yellow amorphous substance and two colourless crystalline bodies, one melting at 230° C. and the other at 108° C. These last three bodies are analogous to similar ones isolated from gutta-percha. The portion of the gum soluble in hot alcohol is not saponifiable, and cinnamic acid cannot be separated from it. All specimens of true gutta-percha, however, do not yield cinnamic acid. A crystalline substance, which readily oxidized in the air, was separated from the chloroform solution. This substance resembled gutta, and an analysis indicated $C_{10}H_{18}$ as the most probable formula for it. A second crystalline substance was also obtained from the chloroform solution, and was found to be analogous to the albanan of gutta-percha. From the above it will be seen that the constituents of balata bear considerable resemblance to those of gutta-percha. W. P. S.

Exudation of Nitro-glycerin from Cartridges. C. N. Hake. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 915.)—An examination of some cartridges, on the outside wrappers of which were observed minute droplets of nitro-glycerin, has shown the exudation to be due to the quality of the paper used for the wrappers, which was of an absorbent nature, gave 1·22 per cent. ash, and acquired a yellowish-brown tint after immersion in milk of lime for four hours, and a dark-blue colour when placed in an aqueous solution of ferric chloride and potassium ferricyanide for five minutes, reactions which indicate the presence of a large proportion of ligno-cellulose. The treatment of such paper with sulphuric acid during manufacture causes charring of the woody matter, thus forming small holes in the finished product, and this porosity of the wrappers explains the mode of the exudation of the explosive folded therein. W. H. S.

The Detection of Traces of Carbon Monoxide in Air. H. Dejust. (*Comptes Rend.*, 1905, cxl., 1250 ; through *Bull. Soc. Chim.*, 1905, xxxiii., 1019, 1020.)—Silver oxide (Ag_2O) is reduced to metallic silver by carbon monoxide, which is converted into carbon dioxide, the heat of the reaction being sufficient to raise the temperature of the mass to 130° C. The reduction also takes place when the silver oxide is dissolved in ammonia solution, the liquid being darkened by the smallest trace of carbon monoxide. For a determination of that gas in the atmosphere the solution is allowed to fall drop by drop into the vessel containing the air, and the tint produced compared with those of standards corresponding to 1 part of carbon monoxide in 1,000, 5,000, and 10,000 parts of air respectively. C. A. M.

The Origin of the Water obtained in the Combustion of Certain Substances. J. A. Muller. (*Bull. Soc. Chim.*, 1905, xxxiii., 953, 954.)—Certain cyanides, such as sodium carbonylferrocyanide, precipitated by alcohol from its aqueous solution, and even potassium ferrocyanide dried at 120° to 130° C., invariably yield a small amount of water in combustion, although presumably free from hydrogen. The author describes experiments which prove that this is due to water of hydration still retained by cyanides after being dried at 120° to 130° C. He also shows that if a long thin indiarubber tube be used for the introduction of the dry air or oxygen into the combustion-tube, moisture may be absorbed from the atmosphere through the rubber. In one experiment, for instance, the amount of moisture absorbed by the sulphuric acid tubes was 0·0034 gram, the volume of dry air that had passed through the tube during seven hours being 2·2 litres. The rubber tube was 35 cm. in length, 5 mm. in diameter, and 1 mm. thick. C. A. M.

The Determination of Aluminium in the Ash of Plants. H. Pellet and C. Fribourg. (*Ann. de Chim. anal.*, 1905, x., 373-381.)—Comparative determinations by different methods, with mixtures of salts containing known quantities of alumina, have led the authors to give the preference to the following modification of Carnot's method: A known volume of the solution in hydrochloric acid, corresponding to 1 or 2 grams of the ash, is nearly neutralized with ammonia, the iron oxidized by means of a few particles of potassium chlorate, and the excess of chlorine expelled. Two grams of sodium or ammonium phosphate are next added, so as to have sufficient phosphoric acid to precipitate all the aluminium, after which 10 grams of ammonium thiosulphate are introduced, then 15 grams of acetic acid, and the liquid boiled for fifteen minutes. The precipitate is collected, washed with 300 to 350 c.c. of boiling water, dried, and ignited; and its weight, multiplied by 0·4185, gives the amount of alumina, to which should be added 0·001 gram to correct for the solubility of the aluminium phosphate in the washing water. In the author's experiments the reagents by themselves yielded a deposit of 0·001 gram, which amount was accordingly deducted in the actual determinations. The results quoted are in close agreement with the theoretical figures. C. A. M.

INORGANIC ANALYSIS.

Volumetric Methods for estimating Copper. G. Fernekes and A. A. Koch. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1224.)—The authors have examined a number of volumetric methods and the gravimetric thiocyanate method, using pure copper sulphate solution, and, in some cases, testing the method on chalcopyrite as well. The *cyanide* method did not give good results. The *potassium iodide* method gave excellent results both with pure copper sulphate solution and with chalcopyrite (in the analysis of which, however, the copper was first separated by aluminium before the titration). A fairly large excess of potassium iodide must be used; thus for 0·0038 gram copper in 100 c.c. solution, at least 1·5 to 2 grams of potassium iodide must be added, whilst for 0·0939 gram copper 2·5 gram potassium iodide are necessary. Arsenic, antimony, tin, cadmium, zinc, and aluminium salts do not interfere with the titration. Lead and bismuth render the end-point somewhat uncertain, but otherwise do not interfere. The *ferrocyanide* method gave fairly good results, especially when the titrations were carried out at 100° C., but the method is applicable only to pure copper solutions. The *gravimetric thiocyanate* method gave excellent results with pure copper solutions, but very low values for chalcopyrite. *Volhard's volumetric thiocyanate* method gave good results with pure copper solutions, but the method is inapplicable to copper ores containing arsenic, as this forms a precipitate with the silver nitrate used to titrate the excess of thiocyanate. *Garrigue's acidimetric method*, in which the thiocyanate precipitate is decomposed with an excess of N sodium hydroxide solution, the excess being afterwards titrated, gave good results for pure copper solutions. The method of *Guess* (ANALYST, 1902, xxvii., 303), in which the thiocyanate precipitate is decomposed with sodium hydroxide, and the sodium thiocyanate formed titrated with potassium permanganate, gave poor results both with pure copper solutions and with chalcopyrite. Equally unsatisfactory were the results obtained in both cases by the use of *Meade's* method (*Journn. Amer. Chem. Soc.*, 1898, xx., 610) in which the cuprous thiocyanate is decomposed as before, the cuprous hydroxide dissolved in ferric chloride or sulphate, and the ferrous salt formed titrated with permanganate. A. G. L.

A Method for the Detection and Determination of Arsenic and Antimony in the Presence of Organic Matter. F. A. Norton and A. E. Koch, (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1247.)

—The authors find that arsenic and antimony are readily obtained free from organic matter by digestion of the tissues, etc., containing them with concentrated sulphuric acid in a Kjeldahl flask. For 5 grams of tissue about 20 c.c. of sulphuric acid is necessary. If the quantities of arsenic or antimony are so small that much larger quantities must be taken, the tissues are extracted in one of the usual ways, and the extract treated with sulphuric acid, 20 grams of which suffice in this case for 100 grams of organic matter. It is found that gentle boiling for one hour with one-sixth hydrochloric acid will extract about 80 per cent. of the arsenic and 90 per cent. of antimony. For quantitative work, the Peterson and Haines modification of Gautier's method gives good results for arsenic, whilst Fresenius and Babo's method is to be preferred for antimony. The residue from the sulphuric acid treatment may be directly titrated with iodine, after neutralization, or, in the case of arsenic, used for the mirror test. Gravimetric methods usually gave somewhat high results. A. G. L.

Iodometric Determination of Aluminium in Aluminium Chloride and Aluminium Sulphate. S. E. Moody. (*Zeits. Anorg. Chem.*, 1905, xlvi., 423.)

—The method described consists in treating the aluminium salt with potassium iodate and iodide, and titrating the iodine liberated according to the equation:

$$Al_2(SO_4)_3 + 5KI + KIO_3 + 3H_2O = 2Al(OH)_3 + 3K_2SO_4 + 6I.$$

For a determination, 25 c.c. of neutral and approximately $\frac{N}{10}$ aluminium solution are heated in a Voit flask with 10 c.c. of neutral potassium iodate solution (containing 30 grams per litre) and 1 gram of potassium iodide for fifteen to twenty-five minutes, or until the liquid has become almost colourless, a stream of hydrogen being passed through the flask at the same time, and the iodine being collected in potassium iodide solution placed in a Drechsel wash-bottle. At the end of the distillation, the iodine in the receiver, as well as that in the flask, is titrated with thiosulphate. Working with quantities of alumina of about 0·04 gram, the error in the test-analyses given varies from -0.0003 to -0.0009 gram. The presence of ammonium salts leads to high results, as these also liberate iodine from a mixture of iodide and iodate. If the receiver contains sufficient acid to combine with the ammonia liberated on boiling, the amount of iodine theoretically equivalent to the ammonium salt is obtained.

A. G. L.

The Estimation of Zinc. H. Nissenson and W. Kettembeil. (*Chem. Zeit.*, 1905, xxix., 951.)

—A long critical paper on volumetric and gravimetric processes, of which only the conclusions can be given. For details, reference must be made to the original paper. Comparison figures for four methods are shown—the sulphuretted hydrogen method, electrolysis, Schaffner's method, and the ferrocyanide method—the analyses being chiefly of various blendes. The figures are strikingly concordant for all methods.

The electrolytic method is, the authors consider, not a possible one in most places, as electric installations are rare in works. The two volumetric processes give equally good results, and it is difficult to choose between them. As regards the ferrocyanide method, the authors did not find that the nature of the oxidizing agent had any influence. They consider that ammonium molybdate or uranium nitrate can be employed indifferently as indicator. The Schaffner method is best carried out in the usual way, as detailed in the paper, and not by using the Belgian 2½-gram process. E. K. H.

The Caustic Soda Method of Determining Molybdenum in Steel. George Auchy. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1240.)—The author discusses objections brought against his caustic soda method (ANALYST, 1902, xxvii., 205) by Cruser and Miller (ANALYST, 1904, xxix., 268), whose method he believes to give correct results only because two errors balance each other. He has slightly modified his own method, however, as follows: 0·8 gram of the sample is dissolved in nitric acid, the solution evaporated to dryness, the residue boiled with 25 c·c. strong hydrochloric acid, and evaporated to fuming with 10 c.c. dilute (3 : 1) sulphuric acid. After taking up with 50 c.c. of water, the solution is poured gradually and with shaking into 100 c.c. of sodium hydroxide solution (1 lb. dissolved in 2,100 c.c. of water) contained in an 8-ounce flask provided with a mark at 200 c.c.; the whole is diluted to the mark, filtered, and 100 c.c. of the filtrate are acidified with 15 c.c. of strong sulphuric acid, reduced with zinc, and titrated with permanganate. If the caustic soda used is fresh, a blank must also be made in the same way, using 0·8 gram of a steel free from molybdenum.

Vanadium, if present, is probably best determined by Glasmann's method (*Ber.*, xxxviii., 600), which depends on reducing the vanadium in one portion by zinc to V_2O_2, and in another by magnesium to V_2O_3. Tungsten, if present, is separated as trioxide; the small amount of molybdenum carried down with it is best determined by adding molybdenum in known amount to tungsten steels free from molybdenum and determining the latter. According to the author, cases of steels containing both tungsten and molybdenum are not likely to occur. A. G. L.

Estimation of Silicon in Different Kinds of Steel. H. Rubricius. (*Chem. Zeit. Rep.*, 1905, xxix., 277.)—The method proposed by the author is as follows: 5 grams of steel shavings are dissolved in 40 c.c. of diluted sulphuric acid (one part H_2SO_4 to two parts of water) at 50° to 60° C. in a tall beaker. After the frothing and evolution of gas have ceased, the sides of the beaker are washed down with very little water, and the liquid heated on a wire gauze. A separation of iron salts ensues, but the liquid can be boiled down, without spitting, until only sulphuric acid remains as liquid (quarter of an hour). The residue is then diluted with 100 c.c. of water, a few c.c. of hydrochloric acid are added, and the liquid boiled in order to dissolve any unattacked steel; another 100 c.c. of water is then added, the liquid filtered, and the carbon containing silica washed with water and hydrochloric acid, and the filter and contents ignited in a platinum crucible.

In cases where the shavings are large, or the steel contains chromium or nickel, the treatment with sulphuric acid must be repeated before passing on, and the addition of hydrochloric acid should also be repeated several times to insure complete solution of the steel. E. K. H.

Note on the Gravimetric Determination of Sulphur in Iron and Steel. Charles R. McCabe. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1203.)—In order to avoid the escape of unoxidized sulphur during solution, the author dissolves 5 grams of the steel in a 1-litre flask, closed by a cork carrying a tap-funnel and a narrow tube about 18 inches long, drawn out to a point at its upper end. Through the funnel 50 c.c. of strong nitric acid are gradually introduced in the flask. To complete solution, the flask is finally heated gently. The solution is then evaporated just to dryness in a dish, and the residue dissolved in 40 c.c. of hydrochloric acid. The solution is evaporated nearly to dryness, diluted, and filtered. The filtrate is heated to boiling, 10 c.c. of a 10 per cent. barium chloride solution are added, and the whole is evaporated till crystals of ferric chloride begin to form, when 175 c.c. of cold water are added. This last evaporation is designed to minimize the solvent action of ferric chloride solution on barium sulphate. After standing for six hours at room-temperature, or two hours in running water, the precipitate is filtered off and ignited. Results obtained in this way on foundry irons are practically identical with those given by the evolution method. A. G. L.

The Electrolytic Determination of Mercury with the Use of a Rotating Anode. Ralph O. Smith. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1270.)—Using a rotating platinum spiral as anode, and a platinum dish as cathode, the author found that 0·2560 gram of mercury, as mercurous nitrate, could be completely deposited, in a coherent form, from a total volume of solution of 115 c.c., containing 1 c.c. of free nitric acid (specific gravity 1·17), the current used being 7 ampères (per 100 sq. cm.) at 6 to 7 volts, and the time required being twenty minutes (*cf.* Exner, ANALYST, 1903, xxviii., 328). Very good results were also obtained when alkali sulphide solutions were used. This latter method is especially convenient for the analysis of cinnabar, and is carried out by boiling 0·5 gram of the powdered ore repeatedly with sodium sulphide solution, using 60 c.c. of 1·06 specific gravity in all, washing to a total volume of 115 c.c., and electrolysing for twenty minutes, using a current of 6 ampères (per 100 sq. cm.) and 5 to 7 volts. In six experiments made in this way on one ore, the quantity of mercury found varied from 46·40 to 46·46 per cent., as against 46·26 per cent. given by the distillation method. The total time taken for an analysis is only ninety minutes. The dish used as cathode loses in weight to a slight extent after each experiment, as a little platinum black is formed, which must be removed by means of sand. A. G. L.

The Rapid Determination of Lead Electrolytically. Ralph O. Smith. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1287.)—The author shows that lead may be rapidly determined as dioxide, using a rotating platinum spiral as cathode and a platinum dish as anode. The volume of the electrolyte was 115 c.c., the anode surface 100 square cm.; the current used was 10 to 13 ampères at about 4 volts. Under these conditions 0·25 gram of lead require fifteen minutes, and 0·5 gram twenty-five minutes for complete deposition. In the first case the quantity of lead found is consistently too high by about 0·0005 gram, and in the latter by about 0·0017, the deposits being dried at 230° C. The author suggests that the high values are due to water retained mechanically, in spite of the fact that the deposits were dried to constant weight. A. G. L.

The Application of the Rotating Cathode to the Electrolytic Determination of Cadmium in Solutions of Cadmium Sulphate and Chloride, with an Appendix to the Same. Charles P. Flora. (*Zeits. Anorg. Chem.*, 1905, xlvii., 1, 13, and 20; *cf.* ANALYST, 1905, xxx., 216.)—The author has made a long study of the application to the determination of cadmium of the rotating cathode (consisting of a platinum spiral revolving 230 or 560 times per minute). Using cadmium *sulphate*, very exact results were obtained when solutions were employed containing (*a*) free sulphuric acid, (*b*) sodium acetate and acetic acid, and (*c*) potassium cyanide. The conditions of working were: (*a*) Cadmium, 0·11 to 0·22 gram; dilution, 45 to 50 c.c.; sulphuric acid, 15 drops of (1 to 4) acid; time, fifteen minutes; current, $N.D._{100}$, 1·5 to 12·0 ampères, at 8 to 12 volts. (*b*) Cadmium, 0·15 gram; dilution, 60 to 65 c.c.; potassium sulphate, 0·2 to 1 gram; sodium acetate, 0·4 to 2 grams, with or without a few drops of acetic acid; time, ten to twenty minutes; current, 2·4 to 3·75 ampères at 7·5 to 12 volts. (*c*) Cadmium, 0·15 gram; dilution, 60 to 65 c.c.; sodium hydrate, 1·0 to 1·5 gram; potassium cyanide, 0·5 to 1 gram; time, thirty to thirty-five minutes; current, 7·5 to 13·5 ampères at 8 volts. Not quite so good were the results obtained with solutions containing urea, formaldehyde, acetaldehyde, pyrophosphates, phosphates, tartaric acid, or formic acid. Bad results were given by solutions containing oxalates, oxalic acid, ammonium tartrate, or potassium formate.

Using cadmium *chloride*, good results were obtained with solutions containing (*a*) sulphuric acid, (*b*) potassium cyanide, (*c*) pyrophosphates, the conditions being: (*a*) Cadmium, 0·1 gram; dilution, 45 c.c.; sulphuric acid, 10 drops of (1 to 4) acid; time, fifteen to twenty-five minutes; current, 6 to 9 ampères at 7·8 volts. (*b*) Cadmium, 0·13 gram; dilution, 65 c.c.; sodium hydrate, 1·0 gram; potassium cyanide, 1·5 gram; time, thirty-five to forty minutes; current, 12 ampères at 7·8 volts. (*c*) Cadmium, 0·13 gram; dilution, 45 c.c.; sodium pyrophosphate, 9·5 grams; ammonia, 1 c.c. of concentrated, or, sulphuric acid, 12 drops of (1 to 4) acid, or, phosphoric acid, 15 drops of 1·7 specific gravity, or, hydrochloric acid, 15 drops of (1 to 4) acid; time, fifteen to forty-five minutes; current, 1·5 to 3·0 ampères at 78 (? 7·8) volts. The other solutions mentioned above gave inferior, or altogether negative results.

Using cadmium *nitrate*, good results were obtained with cyanide solutions under the following conditions: Cadmium, 0·1 gram; dilution, 60 c.c.; potassium cyanide, 0·7 to 1·5 grams; sodium hydrate, 0·5 gram; time, thirty-five to fifty minutes; current, 7·5 to 12·0 ampères at 7·6 to 7·8 volts. Other solutions gave poor results. From liquids containing 1 per cent. or more of free nitric acid cadmium cannot be separated at all electrolytically. A. G. L.

The Electrolytic Determination of Cadmium with the Use of a Rotating Anode. Alice L. Davison. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1275.)—Good results have been obtained by electrolysing various cadmium solutions, using a rotating anode. The cadmium was weighed out in the form of sulphate in each case, the dilution being 90 to 125 c.c., so as to give a cathode surface of about 100 sq. cm. The distance between the electrodes was about 1 cm. Before the electrolysis, the liquid was always heated until bubbles appeared on the bottom of the dish. The best conditions of working are given in the following table:

Cadmium taken (Gram).	Electrolyte.	$N.D._{100}$ Ampères.	Volts.	Speed (Revolutions per Minute).	Time (Minutes).	Error (Gram).
0·5760	0·5 c.c. H_2SO_4(1 : 10)	4	14	630	8	+ 0·0002
0·2867	0·5 c.c. H_2SO_4 + 2 c.c. NH_4OH after five minutes	3	13	630	6	+ 0·0003
0·2887	5 + 6 c.c. NH_4OH + 10 c.c. H_2SO_4(1 · 10)..	5	6	580	5	+ 0·0002
0·2898	1·2 gram Na_2CO_3 + 5 c.c. HCO_2H (specific gravity 1·06)	5	8 to 9	580	4	nil.
0·2898	5 c.c. NH_4OH + 10 c.c. HCO_2H	5	6	580	3	+ 0·0006
0·3984	3 grams $NaC_2H_3O_2$ × 0·25 c.c. $HC_2H_3O_2$ ..		9	580	4	+ 0·0003
0·3575	3 grams $NH_4C_2H_3O_2$ + 3 c.c. $HC_2H_3O_2$ ..		7	580	4	− 0·0002
0·4568	1 gram NaOH + 3 grams KCN		5½	580	20	− 0·0007
0·4568	10 c.c. NH_4OH + 5 c.c. lactic acid ..		7½	580	10	− 0·0024
0·4045	1 gram Na_2CO_3 + 5 c.c. lactic acid ..		9½ to 11	700	10	− 0·0003
0·4040	15 c.c. NH_4OH + 5 c.c. succinic acid (5 per cent.)		11 to 12	700	10	+ 0·0003
0·4040	1 gram succinic acid neutralized by NaOH		9 to 11	700	5	+ 0·0001
	SEPARATIONS.					
0·2660	0·25 gram Mg + 0·2 gram Na_2CO_3 + 12 c.c. HCO_2H (specific gravity 1·06)	5		580	10	− 0·0002
0·2650	0·25 gram Fe (as $FeSO_4$) + 14 grams KCN + 2 grams NaOH..	5 to 5½		700	25	+ 0·0002

From nickel or cobalt, cadmium could not be separated in this way. A. G. L.

The Use of the Rotating Anode and Mercury Cathode in Electro-Analysis. I. Lily G. Kollock and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1255.)—It has been found that a number of metals can be rapidly and conveniently deposited electrolytically, using a rotating anode and mercury cathode. The decomposition cell used is a test-tube 3·5 cms. in diameter and 7·5 cms. in height. A platinum wire, 2 cms. in length, is pushed through the flattened bottom of this tube for a distance of 0·5 cm. Connection is made by placing the tube on a copper plate. The anode is a platinum wire 7·5 cms. long and 1 mm. thick, coiled into a flat spiral 1·5 cms. in diameter. It is rotated at from 100 to 1,800 revolutions per minute. As cathode, 40 to 50 grams of mercury are used. The mercury is very carefully washed with alcohol and ether, and dried in a desiccator before weighing. The distance between the electrodes is 0·5 cm. when the volume of the solution is 5 c.c.; otherwise it is 1 cm. The tube should be covered, and the cover-glasses may be washed down occasionally; but this is generally done by the steam given off owing to the high currents used. When an electrolysis is finished, the anode is stopped, the liquid syphoned off as usual, washing with distilled water until the current is interrupted, after which the mercury is washed with alcohol and ether, dried in a desiccator, and weighed. The same quantity of mercury may be used for two determinations, except for chromium. The best conditions of working for different metals (as sulphates) are summarized in the following table:

Metal.	Amount taken (Gram).	Volume of Liquid (c.c.)	H_2SO_4 added.	Ampères.	Volts.	Revolutions per Minute.	Time (Minutes).	Maximum Error (Gram).
Zinc ...	0·4050	10	0·4 c.c.	5	7	640	5 to 8	−0·0008
Copper ...	0·3945	5	0·2 c.c.	2 to 5	6 to 9	1,200	5	±0·0001
Nickel ...	0·4802	7	0·25 c.c.	6	5 to 6	1,200	7 to 9	−0·0001
Cobalt ...	0·3545	10	0·25 c.c.	5	6	1,200	10	±0·0005
Chromium	0·1190	5 to 15	3 drops	4 to 5	6	400	6	−0·0007
Iron ...	0·2750	5	3 drops	3 to 4	7	520 to 920	7	+0·0005

A. G. L.

The Action of Certain Metals on Molybdenum Salts in the Presence of Sulphurous Acid. G. Dumitrescu. (*Bull. Pharm. Chim. de Roumanie*, 1905, 69; *Ann. de Chim. anal.*, 1905, x., 363.)—On adding a fragment of zinc and a drop of ammonium molybdate solution to a solution of sulphurous acid, a blue coloration, disappearing after some time, is produced. If tin be used in place of zinc, the blue colour is more pronounced, though equally unstable, whilst copper and lead give stable blue colorations. The reactions with mercury and bismuth are very slight, and only take place after some time. By omitting the sulphurous acid, very fugitive green colorations are obtained with zinc, magnesium, iron and lead, the reaction being more sensitive when the metals are used in the form of powder. C. A. M.

The Determination of Phosphoric Acid as Ammonium Phosphomolybdate. Second Paper. Gregory Paul Baxter and Roger Castle Griffin. (*Amer. Chem. Journ.*, xxxiv., 204.)—The authors have confirmed the results set forth in their first paper (*Amer. Chem. Journ.*, 1902, xxviii., 298; ANALYST, 1903, xxviii., 24)—namely, that the composition of ammonium phosphomolybdate varies with the excess of precipitant used, and with other conditions. Phosphorus can only be accurately determined from the weight of the phosphomolybdate if known quantities of reagents are used and suitable corrections are made. The precipitation must always be made by pouring the phosphate solution into the molybdate, otherwise serious variations in the composition of the precipitation may occur. Occlusion of excess of molybdic acid or ammonium molybdate is also favoured by working at any temperature higher than the ordinary room temperature. The ammonium in the precipitate is readily replaced by potassium in the presence of potassium salts, but the reverse change takes place just as easily. The precipitate is also shown to possess the formula $(NH_4)_2HPO_4.12MoO_3$, and not $(NH_4)_3PO_4.12MoO_3$, as is usually assumed. On heating to 300° C., however, with ammonium nitrate, it passes almost completely into the triammonium salt.

A. G. L.

Columbates (Niobates). M. Hume Bedford. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1220.)—The following method was worked out for the separation of niobic acid and tungstic acid in the analysis of niobotungstates. Mercurous nitrate and a slight excess of nitric acid are added to the solution containing niobic and tungstic acids. Freshly precipitated mercuric oxide is then added, and the whole boiled for five minutes. As soon as it has settled, the precipitate is filtered off, washed with boiling water, and ignited, a total weight of $Nb_2O_5 + WO_3$ being thus obtained. The oxides are then fused with potassium carbonate; sodium carbonate should not be used, as the sodium salts are less soluble than the potassium salts. The melt is dissolved in 150 c.c. of water and an excess of ordinary magnesia mixture added. After standing for several hours, the precipitate is filtered off, washed with magnesia mixture, and ignited. The residue obtained is fused with potassium hydrogen sulphate, and the melt boiled with water, after which the insoluble matter is filtered off, washed, ignited, and weighed as Nb_2O_5. The weight of WO_3 is obtained by difference. In five test analyses given, with quantities of Nb_2O_5 varying from 0·15 to 0·5 gram, and of WO_3 from 0·1 to 0·15 gram, the largest error was −1·06 per cent. on the Nb_2O_5, the error in the other four analyses being much smaller.

A. G. L.

The Action of Sulphides on Nitro-prussides. F. Virgil. (*Ann. Soc. Espan. de Fis. y Quim.*; through *Ann. de Chim. anal.*, 1905, x., 363.)—The blue coloration obtained on adding a soluble sulphide to a soluble or insoluble nitro-prusside is due to the formation of an addition compound. If an excess of a soluble nitro-prusside be allowed to act upon a soluble sulphide in the presence of free alkali there is a red coloration, which the author attributes to the simultaneous formation of the above-mentioned blue compound and the yellow substance produced by the action of an alkali or alkaline earth on the nitro-prusside.

C. A. M.

Free Acid in Superphosphate. L. Schucht. (*Chem. Trade Journ.*, 1905, xxvii., 309.)—The author points out the important part played by the free acid in superphosphate, and recommends its estimation by extraction with cold acetone and titration of the dissolved phosphoric acid. Five grams of the superphosphate are placed on a Büchner quick filter and extracted ten times with neutral acetone, which is rapidly drawn off with a filter-pump, about 75 c.c. being used in all. To the filtrate is added 400 c.c. water and some neutral potassium oxalate and sodium chloride, and the liquid then titrated with $\frac{N}{2}$ soda, using methyl orange as indicator.

W. H. S.

The Purification of Arsenical Sulphuric Acid. O. Gasparini. (*Rassegna Mineraria*, 1905, 172; *Ann. de Chim. anal.*, 1905, x., 357.)—Sulphuric acid of 40 to 70 per cent. strength can be completely freed from arsenic by means of a continuous current of 50 to 100 ampères per unit at the anode and 0·5 to 2 ampères at the cathode. The latter consists of a large strip of platinum, whilst the anode is platinum wire. A temperature of 40° to 60° C. and slight agitation accelerate the deposition of arsenic on the cathode.

C. A. M.

APPARATUS.

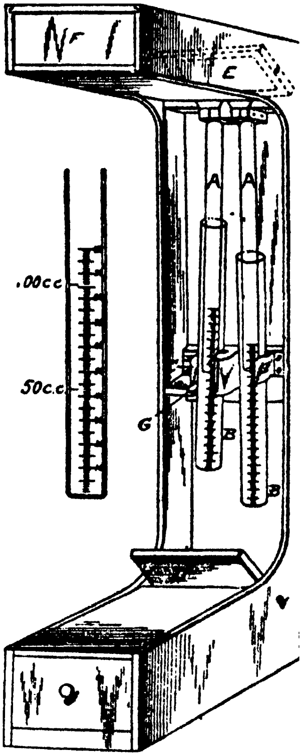
A Strong, Sterilizable Dialysing Membrane. Hibbert Winslow Hill. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1058.)—The membranes are made by soaking silk in gelatine solution, hardening the gelatine by immersion in formaline, and subsequently removing the formaline by washing or steaming. The silk used should be a good, close-meshed, and rigidly-woven, such as white taffeta. If bags or cylinders are made of it, the seams should be doubly or trebly sewn—best with a sewing-machine. The silk is first dipped for ten to fifteen minutes in a 20 to 30 per cent. gelatine solution at 100° C.; it is then lifted out by means of safety-pins attached to its edges, and allowed to drain thoroughly. It is next immersed in ice-cold water for five to ten minutes, in order to allow the gelatine to set, after which it is tested for any leaks by filling it with ice-cold water. Should any flaws be found, the whole process must be repeated. With ordinary care, however, there should be no flaws, and the gelatinized fabric is placed in ice-cold 40 per cent. formaline, and left for about two weeks, the whole being kept during this time in an ice-chest. To remove the formaline, the membrane is then washed in running water or boiled or steamed, after which it is ready for use. Membranes so prepared can be repeatedly sterilized by boiling or steaming under pressure, and dialyse well. If left to dry, they become somewhat brittle, but regain their softness and pliability as soon as they are immersed in water.

A. G. L.

A Simple Colorimeter for General Use, with Remarks on Colorimetric Methods and Apparatus. Oswald Schreiner. (*Journ. Amer. Chem. Soc.*, xxvii., 1192.)—After a discussion of colorimetric methods in general, the author describes the instrument in use at the Bureau of Soils, United States. It consists essentially of two graduated tubes, the height of the liquid in each of which can be altered by moving two other "immersion tubes." The column of liquid below the immersion tube is viewed by looking down this tube. Both unknown solution and standard always being made up to the same volume, if, when the two appear of exactly the same colour, R is the reading of the standard solution of strength S, and r the reading of the unknown solution of strength s, then—

$$s = \frac{R}{r} S.$$

If standard glass slides are substituted for the standard solution, the constant of the slide C is substituted in the above equation for the product RS. These glass slides are especially useful for checking the standard solutions from day to day.



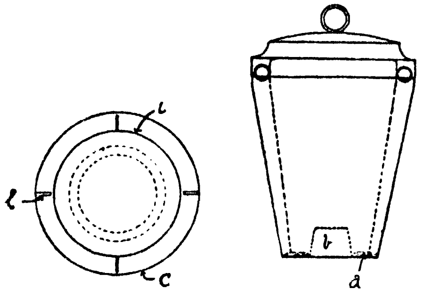
In the figure shown, B, B, are the comparison tubes. They are 26 cm. long, with an inside diameter of 27 mm. The internal bottoms must not be appreciably convex or concave; the outside bottoms are ground and polished, so that the ground surface is slightly larger than the internal diameter of the tube. Each tube is provided with a vertical scale of 100 divisions of 2 mm. each, beginning at the level of the inside bottom, and is also furnished with a 50 c.c. and a 100 c.c. mark for convenience in making up to a known volume. The immersion tubes A, A, are mounted in blocks of wood sliding in grooves in the frame, so that they can be readily removed for cleaning purposes. They are 26 cm. long, with an outside diameter of 19 mm., ungraduated, but otherwise similar to the tubes B, B. These last are held by the brass clips, C, C, which enable them to be readily raised or lowered. Light is reflected up through the tubes from a sheet of cardboard, or opal glass, D, and after passing through the tubes is again reflected from the mirror, E, to the eye of the observer at F. Of course, the glass of the tubes, as well as that of the mirror, must be as colourless as possible. The tubes themselves are made to diverge slightly, so as to give images closer together. The reading taken is that opposite the bottom of the inner tube.

The exactness of the results obtained increases as the two solutions compared approach each other in strength. The chief error, apart from that due to any "personal equation" of the operator, appears to lie in the faultiness of the tubes used. As it appears to be impossible to obtain tubes having their *inner* bottoms ground, for very exact work other tubes are used, which have a plate of plane glass cemented on at the bottom. The cement never lasts long, however, and hence for ordinary use such tubes are too troublesome. A. G. L.

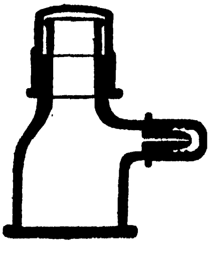
Apparatus for Use in the Gutzeit Test for Arsenic. C. A. Hill and H. S. Collins. (*Chemist and Druggist*, 1905, lxvii., 548.)—The apparatus consists of an ordinary 4-ounce wide-mouthed bottle fitted with an indiarubber bung, through which passes a glass tube. The latter is 200 mm. in length and has an internal diameter of 5 mm.; it is open at both ends, the upper end being slightly widened so that the diameter here is 8 mm., whilst the lower end is drawn out and a small hole is blown in the tube where it is constricted. A roll of lead acetate paper is inserted in the tube, and the mercuric chloride paper is placed over the upper end, being held in position by an elastic band. In each test 10 c.c. of hydrochloric acid (containing arsenic-free stannous chloride), 50 c.c. of water, and 10 grams of zinc are used. Working with 10 grams of the substance to be tested, 1 part of arsenic in 5,000,000 can be detected and determined by the use of the above apparatus. W. P. S.

The use of rubber is highly objectionable.—EDITOR.

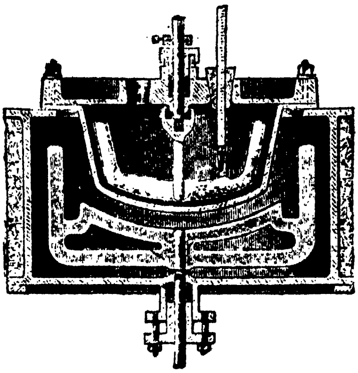
Improved Apparatus for Determining Sulphur and Halogens in Organic Substances. S. S. Sadtler. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1188.)—The method, which has been applied especially to the determination of sulphur in oils, consists essentially in heating the substance with a modified Eschka mixture (1 part of magnesia to 1 part of sodium carbonate) in an inner platinum crucible inverted in an outer and larger one, the latter being provided with a closely-fitting lid. The outer crucible is provided with an indentation (*b*) in its bottom, so as to enable the mass inside to be more strongly heated. Contact between the two crucibles is prevented by a layer of asbestos (*a*) and the platinum rings (*l*) soldered to the inner crucible. For a determination, the substance, if a solid or difficultly volatile liquid, is weighed out directly into the inner crucible; if a very volatile liquid is to be analysed, it is contained in a bulb with capillary tube. The inner crucible is filled with the Eschka mixture, and the outer crucible is inverted over it; the whole is then inverted, and a layer of magnesia about ¼ inch in depth filled in between the two crucibles. After putting on the lid, the crucibles are heated, gently at first, and then strongly. Finally, the inner crucible is removed so as to empty the whole mass into the outer crucible, and any carbon left is burnt off, after which the determination is finished as usual. The method gives results agreeing closely with those given by the Carius method. A. G. L.

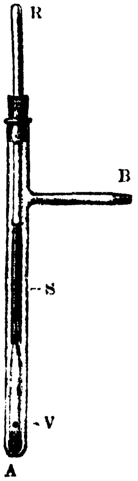


Dropping Bottle for Hydrofluoric Acid. A. Gwiggner. (*Chem. Zeit.*, 1905, xxix., 671.)—The bottle has the shape shown in the illustration, and is made of hard caoutchouc. The side-jet is closed by means of a screw-cap when the bottle is not in use. To obtain drops of acid from the bottle, this cap is removed, and the cap closing the neck slightly unscrewed, so as to admit gradually a small volume of air. The bottle is patented, and may be obtained from W. J. Rohrbecks' Succsr., Vienna. W. P. S.



A Small Heating Apparatus or Boiler with Stirrer. C. Schwalbe. (*Chem. Zeit.*, 1905, xxix., 670.)—The inner basin of the apparatus is of about 250 c.c. capacity, but not more than 100 c.c. of the liquid or liquids to be treated should be placed in it. The air-tight lid is provided with a central opening and stuffing-box, through which passes the spindle of the stirrer. A mercury trap may be placed above the stuffing-box, and a small circular trough is fitted on the spindle under the cover, to catch any oil drops, etc., and to prevent them falling into the contents of the basin. Three other holes are also provided in the lid, one for the thermometer, a second for a reflux condenser, and the third serves for filling the basin. The latter is surrounded by a larger vessel coated with asbestos, or the like. This vessel is provided with a stirrer, and may be filled with any suitable liquid. It is heated by means of a ring-burner (not shown in the figure). The apparatus may be employed for heating together immiscible liquids, distillation under reduced pressure, or the outer vessel may be filled with a freezing mixture, etc., for a variety of cooling operations. It is made of acid-resisting or enamelled ware, and may be obtained from Ehrhardt and Metzger's Successors, Darmstadt. W. P. S.





Automatic Vacuum Regulator.—The object of this apparatus is to enable the operator to maintain a constant pressure when conducting an operation such as distillation under reduced pressure. The apparatus consists of a glass tube, having a side-arm at B, which is connected with the apparatus to be exhausted, and having also at its lower extremity, A, a specially devised valve, V, which is pressed down by the spring S, and kept in position by the adjustable rod R. The joint pressure of the air and of the spring keeps the valve closed until, as air is pumped out of the apparatus, the pressure on the valve is sufficiently diminished to overcome the before-mentioned two pressures, when air is admitted until equilibrium is again restored. By moving the rod up and down, any pressure, within certain limits, may be continuously maintained. The apparatus is made by J. J. Griffin and Sons, Kingsway, London. W. J. S.

A New Burette Holder. A. T. Lincoln. (*Jour. Amer. Chem. Soc.*, 1905, xxvii., 1442.)—The holder consists of a well-seasoned block of wood, ¾ inch thick, 3·5 wide, and 9 inches long. The ends are notched back for about 2 inches, each end then resembling the letter M turned on its side. Against these M pieces the burettes rest in two grooves cut in the face of the block, which secure them in a position parallel to one another. Another groove is cut out, medially, of the back of the block, and into this the rod of the burette stand fits. The burettes are kept in position by means of a piece of springy sheet brass, $\frac{3}{4}$ of an inch wide, and this, extending across the front of the block, serves also as a washer to a thumb-nut which tightens a bolt passing through the board. This bolt is provided with a staple-head which passes round the rod. If necessary, the brass spring may be reinforced with a second shorter brass plate placed in front of it. It is claimed that, with this holder, burettes may be more readily fixed in position, and moved up and down much more easily than with the older form of holders. A. G. L.

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REVIEW.

GMELIN-KRAUT'S HANDBUCH DER ANORGANISCHEN CHEMIE. Seventh Edition.

The seventh edition of this well-known and most comprehensive work on Inorganic Chemistry is in course of publication (Carl Winter, Heidelberg). It is planned to appear within three to four years in about seventy parts, each comprising four sheets (64 pages). Although, unfortunately, one of the editors—Professor A. Hilger, of Munich—died recently, the MS. of the work is virtually completed, and the publishers do not anticipate any delay.

Judging by the first part, comprising oxygen, ozone, and atmospheric air, the new edition will contain, in the most pithy and condensed form compatible with clearness, almost every fact belonging to inorganic chemistry, with countless references to the original publications. O. H.

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THE ANALYST:

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, December 6, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

The Council's nomination of Dr. H. W. Wiley as an honorary member, and certificates of proposal for election to membership in favour of Messrs G. Clarke, jun., C. A. Hill, B.Sc., and H. J. Horton, were read for the second time; and certificates in favour of Messrs. Samuel Dickson, chief assistant to Mr. R. H. Harry Stanger, 2, Broadway, Westminster, S.W.; John Evans, F.I.C., chief assistant to Mr. G. E. Scott-Smith, 67, Surrey Street, Sheffield; Frank Hughes, chemist to the Khedivial Agricultural Society, Cairo; George Patterson, F.I.C., chemist to the Manbré Saccharine Company, Limited, Fulham Palace Road, Hammersmith, W.; and Harry Thompson, consulting and analytical chemist, 5, Bishop Lane, Lowgate, Hull, were read for the first time.

Messrs. C. F. Cross, B.Sc., and C. E. Male were elected members of the Society.

Messrs. C. G. Moor, M.A., and Arthur R. Ling were appointed auditors of the Society's accounts for the year 1905.

The following papers were read: "The Reducing Action of Hydrogen. II. The Estimation of Traces of Arsenic by the Marsh-Berzelius Method, and the 'Insensitiveness' of Zinc," by Alfred C. Chapman and H. D. Law, B.Sc.; and a "Note on the Removal of Arsenic from Hydrochloric Acid for Use in the Marsh-Berzelius Method," by Arthur R. Ling and T. Rendle.

Mr. G. T. Holloway, A.R.C.Sc., exhibited an improved optical arrangement for reading balance graduations.

* * * * *

SURIN FAT.

By J. LEWKOWITSCH, PhD.

(Read at the Meeting, November 1, 1905.)

Through the kindness of Professor W. R. Dunstan, I obtained a sample of fat marked "Minyak surin, from Perak, Straits Settlement." The origin could not be stated. It was added that the fat is probably derived from the seeds of a species of *Palaquium*. After removing some impurities by filtering, the fat was examined with the following result:

Fat.

Free fatty acids...	43·2 per cent.
Specific gravity at 60° C. (water at 60° C. = 1)	0·9021
Solidifying point, commences at	48·9° C.
,, ,, solid at	43·90° C.
Melting-point (capillary tube)	56·1° C.
Saponification value	179·5
Unsaponifiable matter	4·54
Iodine value	42·31 per cent.
Reichert-Wollny value	0·55 ,,

Fatty Acids.

Solidifying point	59·1° C.
Mean molecular weight	284·9
Stearic acid (of melting-point 67·8° C.)	58·2 per cent.

Evidently the fatty acids consist of a mixture of stearic acid and oleic acid only. The extremely high proportion of stearic acid would seem to render this fat a very useful raw material for the candle industry, although the considerable amount of unsaponifiable matter might militate against its use. Possibly the high proportion of unsaponifiable matter is due to careless preparation of the fat.

Although a number of species (eighteen) of the genus *Palaquium* (family of Sapotaceæ), all of which yield guttapercha, have been described, the fats obtainable from them are hardly known. The seeds of *Palaquium Pisang*, Burck (Sumatra), are stated to yield 45 per cent. of a bitter yellowish fat known in commerce under the name "Balam tallow."

From the seeds of *Palaquium oleosum*, Blanco (Sumatra), a white sweetish fat ("Suntei tallow") is expressed. *Palaquium oblongifolium*, Burck (Borneo), is said to yield a hard white fat ("Njatuo tallow"), consisting chiefly of "stearine" (whatever this may mean). It is used in West Borneo as an edible fat. Recently De Jongh and Tromp described the fat from *Palaquium oblongifolium* (which yields the best kind of guttapercha), and gave the following figures:

Yield of fat from seeds	32·5 per cent.
Melting-point of fat	40° C.
Saponification value	201·5
Iodine value	34·3

The specimen examined by them was comparatively fresh, as the percentage of free fatty acid was only 2·1.

I suggest retaining the native name "Surin fat" for this fat, which obviously differs from the fat from *Palaquium oblongifolium*—at any rate, until the proper source has been ascertained.

* * * * *

THE REDUCING ACTION OF HYDROGEN.

II. The Estimation of Traces of Arsenic by the Marsh-Berzelius Method, and the "Insensitiveness" of Zinc.

By ALFRED C. CHAPMAN, F.I.C., and H. D. LAW, B.Sc.

(*Read at the Meeting, December* 6, 1905.)

The urgent need which arose a few years ago for a method capable of detecting and estimating with accuracy minute traces of arsenic caused numerous investigators to devote their attention to the solution of a problem which was soon found to be one of very considerable difficulty. It is unnecessary for us to refer to the numerous methods which were at various times suggested, more especially as the modified Marsh-Berzelius process, with which we are particularly concerned in this paper, is now almost universally employed. The energies of analysts were at first devoted to getting reagents of the high degree of purity necessary, and as the result of much labour zinc and mineral acids were obtained which were either free from arsenic or contained only such infinitesimal traces that perfectly blank results were given in an apparatus capable of revealing with certainty $\frac{1}{500}$ mgr. It soon, however, came to be recognised that freedom from traces of arsenic was not the only qualification which zinc for use in the Marsh-Berzelius flask must possess, since many samples of the presumably pure metal were found to exhibit the property of preventing the reduction of more or less of the arsenic added, and so were worse than useless. One of the first observations made in connection with this aspect of the subject was that the addition of a few drops of platinum chloride to the contents of the Marsh-Berzelius flask was entirely inadmissible, as it had the effect of causing the "retention" of comparatively large quantities of arsenic. One of the authors showed that the same applied in a less marked degree to solutions of copper sulphate. These facts naturally led to an investigation of the effects of depositing traces of certain metals on the surface of "sensitive" zinc, and it was found that whilst some metals—*e.g.*, Au and Pt—exerted a marked retentive effect, others—*e.g.*, Ag and Cu—exhibited this property in a far less degree. At one time it was thought that the influence exerted by the added metallic impurities was in some way connected with their position in the electro-chemical series, but one or two results were opposed to this view, and seemed to show that the true explanation was not by any means so simple. As the experimental difficulties were very great, and as the work involved the expenditure of more time than could be spared, the matter was allowed to drop for the moment. One or two chance observations, however, which seemed to throw a little light on the subject induced the authors to recommence the study of these phenomena, and it was decided to attack the problem in a systematic manner. To this end we studied, in the first place, the effects produced by adding to the Marsh-Berzelius flask containing sensitive zinc, solutions of various metallic salts, the quantities taken varying from 2 grams to 0·1 gram. The following table, in which the numbers represent thousandths of a milligram, will show the results obtained:

Salt added.	Quantity in Grams.	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Flask.
Palladium chloride ...	0·1	50	None	50
,, ,, ..	0·1	500	Practically none	Practically 500
Platinum chloride ...	0·1	500	None	500
,, ,, ...	0·1	1,000	Practically none	Practically 1,000
Nickel sulphate ...	2·0	200	20	180
Cobalt sulphate ...	2·0	200	20	180
Stannous chloride ...	2·0	10	10	—
Lead acetate	1·0	10	10	—
Cadmium sulphate ...	2·0	10	10	—

In addition to the above, experiments were conducted with small quantities of salts of metals incapable of being deposited on the zinc. Thus, we experimented with the sulphates of potassium, aluminium, magnesium, sodium, and ammonium, and found that there was either no retentive effect at all, or else that this was so slight that it might well have been accounted for by traces of impurity.

An inspection of the above table shows that whilst palladium, platinum, nickel, and cobalt all exert a more or less marked retentive effect, cadmium, tin, and lead appeared to be entirely without such action. The "retention" was clearly, therefore, not in any way connected with the position of the added metal in the electro-chemical series, and it became necessary to find some other explanation of these apparently anomalous results. The observation made by one of us on the reducing action of the hydrogen in palladium-hydrogen had clearly shown the insufficiency of the conceptions of active or atomic and inactive or molecular hydrogen to explain various reduction phenomena, and had rendered it evident that the chemical activity of the hydrogen in many of the reactions studied depended very greatly on the source from which it was obtained. Having this aspect of the question in mind, we turned our attention to the work which had been done by Caspari in this direction. The interesting observations of Thorpe, Tafel, Sand and Hackford, Trotman, and Thomson in regard to the varying reducing efficiency of different hydrogen electrodes all supported this view, and appeared to us to furnish an additional reason for an extended and more systematic study of the question.

Before proceeding to give an account of our experiments, we may perhaps be permitted to refer to the following theoretical considerations:

If a rod of zinc be immersed in a dilute solution of a salt of copper, the zinc tends to pass into the solution and drive out the copper. On the other hand, a strip of copper placed in a solution of a zinc salt endeavours to force out the zinc from its solution and cause it to be deposited in the metallic state, and at the same time to form a salt of copper. Whether the zinc or the copper goes into solution depends on which metal exercises the greater force, and the same reasoning applies to every cathion pair. In the case we are considering the solution tension of the zinc is about 1·7 volts, whilst that of copper is much less, and so a deposition of metallic copper might be expected. There is, however, another important factor to be borne in mind—viz., the supertension. It would be expected that every metal should be deposited from the solution of its salts when a potential difference is exerted slightly in excess of the particular solution tension of that metal. As a rule, however, a considerable force (known as supertension) in excess of this has to be applied before the deposition is brought about. Thus, in the case of a mixture of zinc and dilute sulphuric acid, very little hydrogen is liberated, although the solution tension of zinc is about 1·7 volts, and that of hydrogen only about 1 volt, the zinc in this case exerting a retaining effect on the hydrogen equal to about 0·7 volt. Our experiments deal principally with the solution of metals in sulphuric acid, which we will now proceed to study in detail as a particular example of a general law. Each metal exerts a supertension with respect to hydrogen, the largest in our experiments being zinc, and the smallest platinized platinum. If now we deposit a particle of platinum on a piece of zinc placed in dilute sulphuric, we have obtained an outlet for the hydrogen at a low potential, and, consequently, as the solution tension of the zinc is 1·7 volts, and the supertension of the platinum only a very small fraction of a volt, the hydrogen is liberated with greatly increased rapidity. The increased velocity of the evolution we may suppose to be directly proportional, up to a certain point, to the number of molecules present on the surface. The hydrogen obtained from a pure zinc surface will, however, have properties differing entirely from those in the other case. In the first case, where the potential of the hydrogen is high, more difficult reductions may be effected than in the other. These considerations have been applied to the Marsh-Berzelius test for minute quantities of arsenic. In the first place, let us take the simplest case of the solution of pure zinc in sulphuric acid in presence of arsenious oxide. The hydrogen bringing about reduction may be supposed to be in the atomic condition, possessing one free bond and a potential of 1·7 volts. This hydrogen must be in solution, and the amount present at any instant will be constant and independent of the rate of evolution—of course, after the solution in the neighbourhood of the metal has become saturated. On the other hand, the arsenious oxide may be supposed to dissociate, to a small extent, into trivalent ions possessing three free bonds and a certain definite potential, a reduction taking place when these free bonds are satisfied by hydrogen. This may be expressed in the following manner:

$$\mathrm{As_2O_3} \rightleftarrows 2\mathrm{As}{\equiv} + 3\mathrm{O} \qquad \mathrm{As}\_ + 3\mathrm{H} \rightleftarrows \mathrm{AsH_3}.$$

We have now a number of factors influencing the reaction to take place. Suppose the active mass of hydrogen is M_{H} and that of the free arsenic ions M_{As}. After the action has been allowed to take place until as much arseniuretted hydrogen has been driven off as it is possible to obtain, there will be still left in solution a very minute quantity represented by $M_{\mathrm{AsH_3}}$. We shall have now obtained a state of equilibrium depending on our five factors. $M_{\mathrm{AsH_3}}$ must, according to the mass law, be proportional to the product $M_{\mathrm{As}}.M_{\mathrm{H}}$. There is, however, another influence at work, and one overlooked apparently by previous investigators, viz., the potential of the various active ions. Thus the factor M_{AsH_3} must also be proportional to the product of two factors $P_H.P_{As}$ depending on the potential of the hydrogen and of the arsenious ions respectively, or expressed in the form of an equation:

$$M_{AsH_3} \propto M_{As}.P_{As} \times M_H.P_H;$$

or—

$$M_{AsH_3} \rightleftarrows kM_{As}.P_{As} \times M_H.P_H,$$

where k is a constant. The arseniuretted hydrogen will also tend to decompose with some constant force which has been included in the factor k. The amount of arseniuretted hydrogen left in solution may be regarded as constant, or nearly so, at any moment during our experiment. M_H and P_{As} are also constant factors, independent of the condition of the metal, and we may therefore write our equation—

$$M_{AsH_3} \rightleftarrows k,M_{As}.P_H,$$

when k is a constant factor. That is to say, the active mass of the AsH_3 is proportional to the product of the potential of the hydrogen and the active mass of the arsenious ions. Owing to the extreme dilution of the solutions in question this latter also may be considered proportional to the total arsenious oxide present, and we may write finally:

$$M_{AsH_3} \rightleftarrows KAsP_H,$$

where As represents the amount of unreduced arsenious oxide, and K a constant factor. If now the above equation is disturbed by the addition of As—*i.e.*, if the factor As is increased—reduction takes place, or the reaction proceeds from right to left, and the same result will be reached if P_H is made larger. On the other hand, if P_H is made smaller As must be made larger to preserve the equilibrium. In other words, there is always left in solution after each experiment a certain residuum of unreduced arsenious oxide. This is very small when the factor P_H is large, as in the case of pure zinc, and must, we know, be far less than $\frac{1}{500}$ mgr., and probably less than $\frac{1}{1000}$ mgr., as this quantity can with certainty be detected. On the other hand, when P_H is small then the oxide left in solution may be relatively considerable. This is very pronounced in the case of zinc coated with platinum, in which case it may be more than 1 mgr. This phenomenon was observed recently by Sand and Hackford in the case of copper spirals in an electrolytic apparatus, when it was shown that any quantity of arsenious oxide above $\frac{1}{200}$ mgr. could be detected, but nothing smaller. In the above considerations we have been dealing with arsenite solutions, but very little additional difficulty is presented in the case of arsenates. In the latter case we have two processes taking place simultaneously—first, the reduction of arsenate to arsenite, and, secondly, the formation of arseniuretted hydrogen. The first processes must take place with extreme ease, as our experiments with the Marsh-Berzelius apparatus show that in both cases equally good arsenic deposits are obtained. It is extremely probable that similar explanations apply to other reduction process, and it should be in many cases possible to predict results. Some of these we propose to consider in a future communication. The first to draw attention to the phenomenon of supertension was Caspari, who arranged the metals in the following order: Platinum (black), gold, iron, platinum (polished), silver, nickel, copper, palladium, cadmium, tin, lead, zinc, and mercury, the first-mentioned metal having the lowest value, whilst mercury possesses this property in the highest degree. According to our view, this will also represent the order of efficiency of these metals in any reducing action in which they take part, a view of the correctness of which we have obtained ample experimental confirmation. The only exception is palladium, which we have placed along with platinum and gold. Tafel, in a paper on the electrolytic reduction of certain organic substances, placed the metals in the following order: Platinum, silver, tin, copper, mercury, zinc, iron, in which iron is credited with reducing properties equal to those of mercury and lead. This is, however, directly opposed to both Caspari's and our own work, and the point was, therefore, studied in detail, five alloys of iron being prepared and examined; but in each case the result confirmed our view that iron must be placed among the metals of comparatively feeble reducing activity. Iron, however, exhibits a very curious property, which we will proceed to describe. The introduction of a metallic salt into the Marsh apparatus is invariably accompanied by a sudden increased liberation of hydrogen, if the cathion happens to be a metal of low supertension. This, however, did not, as a rule, take place in the case of iron salts until after violent agitation of the solution in the apparatus, but on the addition of a small quantity of arsenious oxide the action became as brisk as in the case of other metals of low supertension. The explanation appears to be that the layer of hydrogen prevents the iron from coming into contact with the zinc, and does not, therefore, permit the formation of an actual couple. Violent agitation, or the addition of an oxidizing agent, however, removes this hydrogen and permits the formation of the couple. A similar phenomenon was noticed in the potential experiments described below. Here it was observed that the potential of the zinc did not drop suddenly on the addition of an iron salt, as might have been expected, but only slowly. When, however, the surface hydrogen was removed from the zinc by means of a little arsenious oxide the iron fell into line with our other experiments. Similar properties were noticed in the case of other metallic salts, but the action was very much less marked. On studying the results of Caspari's experiments on supertension it at once became clear to us that those metals which appeared to have no effect in causing the retention of arsenic, and whose behaviour we had been unable to explain, were precisely those possessed of high supertensions, and we therefore proceeded to study the question in greater detail with this possible solution in our minds. At the outset it became evident that the addition of metallic solutions was not the best way in which to make a comparative study, inasmuch as in many cases the deposited metal separates largely from the zinc, and so ceases to be effective. We therefore decided to work with zinc alloys. To this end small quantities of the metals whose effects were to be studied were alloyed in a graphite crucible with highly sensitive zinc, and the resulting product granulated in the ordinary way. The numbers given below in the second column, and representing the proportions of the added elements, are in most cases only approximate, owing to the difficulties inseparable from the preparation of such alloys; but they, of course, represent in all cases the maximum quantities of the added impurities.

ALLOYS.

Metal added.	Quantity of Metal added to 100 Grams Zinc.	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Flask.
Nickel I. ...	0·1 gram.	30	10	20
Nickel II. ...	0·5 ,,	20	5	15
Nickel III. ...	0·1 ,,	20	10	10
,, ...	0·1 ,,	10	4	6
Cobalt ...	0·1 ,,	30	6	24
,, ...	—	20	4	16
Cobalt II. ...	0·5 ,,	10	2	8
Iron I.	0·1 ,,	30	20	10
,, ...	—	20	2	18
Iron II. ...	0·1 ,,	30	10	20
,, ...	—	20	5	15
Iron III. ...	0·5 ,,	20	2	18
Iron (reduced)	0·5 ,,	30	5	25
,, ...	—	10	2	8
Iron (wire) ...	0·5 ,,	50	10	40
,, ...	—	30	5	25
,, ...	—	10	1	9
Copper	0·1 ,,	30	10	20
,, ...	—	20	2	18
Silver	0·2 ,,	30	12	18
,, ...	—	20	5	15
Platinum ...	0·2 ,,	30	6	24
Sodium ...	5 grams.	10	5	5
Tin	0·5 gram.	10	10	—
Cadmium ...	0·5 ,,	10	10	—

At this point we would call attention to the fact that iron exercises a very marked retentive effect, as this is opposed to certain published observations. We propose to revert to this a little later. From the above results, which entirely confirm those obtained with saline solutions, it will be seen that those metals which are known to possess low supertension values (see Caspari's order above), have in all cases the property of preventing the conversion of arsenious oxide into arsenic hydride, whilst others—*e.g.*, cadmium, tin, and lead—which have high values, exercise no appreciable effect. Very numerous experiments having confirmed the correctness of this explanation of the so-called "insensitiveness" of zinc, it clearly became a matter of interest, as well as of practical importance, to see whether the results we had obtained could not be applied to the conversion of insensitive zinc into the sensitive metal. Since in a zinc-iron couple the hydrogen may be supposed to be liberated largely from the iron particles, it seems reasonable to conclude that other cathions would also be liberated at the same points, or, in other words, form a coating over them, and thus mask their action. To test the correctness of this view, we added cadmium sulphate to the contents of the Marsh-Berzelius flask containing highly insensitive zinc, and found that under these circumstances even such small quantities of arsenic as $\frac{1}{100}$ mgm. could be readily and with certainty detected. In the following table we give examples of this:

Alloy of Zinc with	Salt added.	Arsenious Oxide added.	Arsenious Oxide found.
Iron I.	Cadmium sulphate (2 grams) ...	20	20
		10	10
Iron II. ...		10	10
Iron (reduced)		10	10
Iron (wire) ...		10	10
Silver ..		10	10
Platinum ...		10	10
Copper ...		10	10
Cobalt ...		10	10
Sodium ...		10	10
Nickel I. ...		10	7
Nickel II. ..		10	7

Similar results were obtained with tin and lead, as might have been anticipated from a consideration of the views we have put forward. In the following table experiments with these metals are recorded :

Alloy of Zinc with	Salt added.	Arsenious oxide added.	Arsenious Oxide found.
Iron II.	Stannous chloride ...	10	10
,, ...	Lead acetate ...	10	10
Iron (reduced) ...	Stannous chloride ...	10	10
Iron (wire) ..	,, ,, ...	10	10
,, ...	Lead acetate ...	10	10
Silver	Stannous chloride ...	10	10
	Lead acetate ...	10	10
Platinum	Stannous chloride ...	10	10
,,	Lead acetate ...	10	10
Copper	Stannous chloride ...	10	10
,,	Lead acetate ...	10	10
Cobalt	Stannous chloride ...	10	10
Sodium	,, ,, ...	10	10
,,	Lead acetate ...	10	10
Nickel I.	Stannous chloride ...	10	7
,,	Lead acetate ...	10	7
Nickel II.	Stannous chloride ...	10	10
	Lead acetate ...	10	10

On referring to the above numbers, it will be seen that some of the results with nickel-zinc alloys are low. This appears to be due to the fact that the nickel exhibits a marked tendency to become separated, in the form of minute dark particles, from the zinc during the solution of the alloy; these particles pass into solution in the acid, and subsequently become deposited on the cadmium surface.

Three samples of insensitive zinc, as supplied by chemical dealers, were then experimented with. These, referred to below as "A," "B," and "C," contained the varying amounts of iron as stated. It may be added in this connection that several samples of highly sensitive zinc were found to contain only infinitesimal traces of iron.

	"A."	"B."	"C."
Fe	0·09 per cent.	0·24 per cent.	0·66 per cent.

The following results were obtained when using these three samples in the Marsh-Berzelius flask :

Zinc "A."

	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Flask.
Zinc alone	5	4	1
Zinc + 2 grams cadmium sulphate	5	5	—
	Zinc "B."		
Zinc alone	5	1	4
,, ,, ...	10	5	5
Zinc + 2 grams cadmium sulphate	5	5	—
,, ,, ,, ,,	10	10	—
	Zinc "C."		
Zinc alone	5	1	4
,, ,, ...	10	2	8
Zinc + 2 grams cadmium sulphate	5	5	—
,, ,, ,, ,,	10	10	—

Having thus been successful in overcoming the tendency exhibited by some samples of zinc to retain arsenic in the Marsh-Berzelius flask, we thought it would be interesting, from a theoretical point of view, to see whether other "insensitive" metals capable of being used for the purpose of obtaining hydrogen might not be similarly rendered sensitive. To this end we experimented with two samples of magnesium, and the results are given in the following table :

Magnesium "A."

	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious left in F
Magnesium alone	20	10	10
Magnesium + 0·1 gram nickel sulphate	1000	5	995
Magnesium + 0·1 gram iron sulphate	500	4	496
Magnesium + 0·1 gram cadmium sulphate	20	20	—
Magnesium + 0·1 gram zinc sulphate	20	20	—

MAGNESIUM "B."

	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Flask.
Magnesium alone	10	4	
Magnesium + 2 grams cadmium sulphate	10	10	
Magnesium + 2 grams cadmium sulphate	5	5	
Magnesium + 2 grams stannous chloride	10	10	
Magnesium + 2 grams lead acetate	10	10	

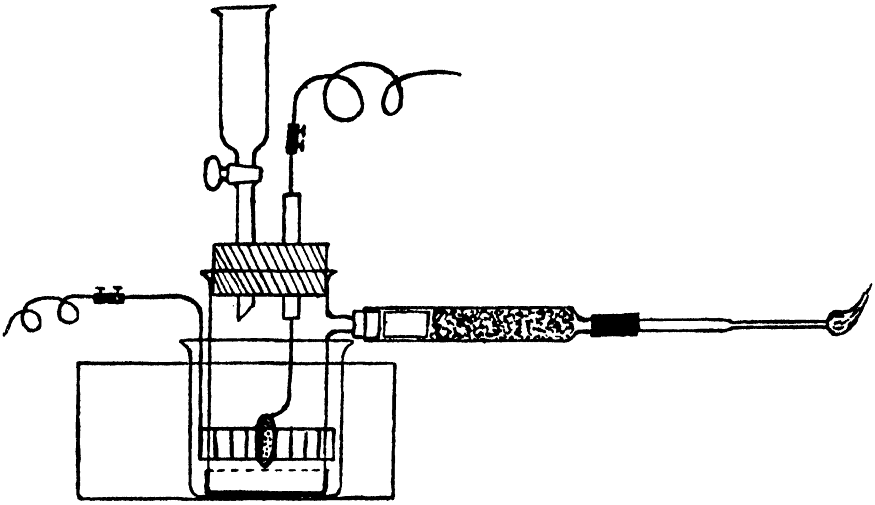
Similar experiments made with calcium and aluminium failed to give results: in the former case, owing to the impossibility of regulating the reaction, and in the latter, owing to the formation of a white deposit in the heated arsenic tube.

In the next place, solutions of arsenic oxide were experimented with in precisely the same manner as the arsenious oxide solutions above mentioned, and the results were found to be in all cases strictly comparable, as will be seen in the following table:

	Arsenic Oxide added (Equivalent to Arsenious Oxide).	Arsenic Oxide found (Equivalent to Arsenious Oxide).	Arsenic Oxide left in Flask (Equivalent to Arsenious Oxide).
Zinc + 2·0 grams nickel sulphate ...	500	50	450
Zinc + 2·0 grams cobalt sulphate ...	500	50	450
Zinc + 0·1 gram platinic chloride ...	1000	—	1000
Zinc + 2·0 grams cadmium sulphate...	10	10	—

Seeing that all the above results are susceptible of explanation on the assumption that the potential of the hydrogen is one of the determining factors, we felt that the study of this question would not be complete without a repetition of some of the electrolytic work which has already been done. This appeared to us to be the more necessary, inasmuch as one or two of our observations are not in agreement with those of previous investigators. The following is a description of the apparatus employed.

The cathode compartment consisted of a wide glass tube 8 cm. deep, having an internal diameter of 3·5 cm., and provided with an outlet tube at the side. The top of this compartment was closed with a rubber stopper through which passed a tap-funnel and a short piece of glass tube to support the cathode. Over the bottom was stretched a parchment diaphragm which was held in position by an elastic band. The side tube was connected by means of a cork with the drying apparatus containing the usual lead acetate paper and calcium chloride. The anode consisted of a platinum ring placed in a beaker slightly larger than the cathode compartment. The whole apparatus was placed in a vessel of cold water to keep the solutions cool during each experiment. As it was necessary to change the cathode from time to time, this was kept in position by means of a sealing-wax joint. In the preliminary work it was found that the best results were obtained with high-current densities and from moderately concentrated solutions of the arsenic compounds. For this reason a cathode having a superficial area of 4 square cm. on both sides was used, and the amount of liquid in the cathode compartment did not exceed 18 c.c. In every case a current of 6 ampères was used, and no particular attention was paid to the voltage, as this varied very considerably with different parchment diaphragms. Two sets of experiments were made, and no arsenic was added until the apparatus had been in action for ten minutes to test the purity of the reagents.



The following results were obtained from arsenite solutions with various cathodes, the acid used being in each case 10 per cent. sulphuric acid by volume:

Cathode.	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Flask.
Lead	10	10	
Cadmium	10	10	
Tin	10	10	
Copper I.	100	10	90
Copper II.	50	2	48
Silver	50	10	40
Nickel	40	20	20
Platinum (black)	1,000	0	1,000
” ”	10,000	Large	
Iron	50	5	45

Similar electrolytic experiments to the above were next made, using solutions of arsenic oxide, and in all cases smaller mirrors were obtained than those yielded by corresponding amounts of arsenic in the arsenious state.

ARSENATES.

	Arsenates added corresponding to Arsenious Oxide.	Arsenic found.	Arsenic left in Flask.
Cadmium	10	7	3
Lead	10	7	3
Copper II.	70	10	60
Nickel	40	10	30
Platinum (bright) ...	40	2	38
„ (black)	10,000	Large	—

The above results with cathodes of platinum, lead, cadmium, tin, nickel, and copper confirm the view we have expressed above. It will be seen that with the iron electrode we have a considerable retention, just as with the solutions and alloys of that metal. We make special reference to this point, inasmuch as Tafel, and Sand and Hackford working conjointly, have credited an iron electrode with a reducing efficiency comparable with that of lead and zinc. When using copper cathodes the following phenomena were observed, which we think worthy of being recorded:

The cathodes of this metal invariably became dulled after a few experiments, owing, probably, to the deposition of metallic arsenic on the surface; but at the same time the electrode became more and more sensitive, although no mirror could be obtained in a blank run for half an hour. The following experiments were therefore made: Two copper cathodes were allowed to act in electrolytic cells, one containing a large excess of arsenious oxide and the other a similar quantity of arsenic acid for one hour, when both had become covered with elementary arsenic in a non-reducible form. The following experiments were then made:

ARSENIOUS OXIDE.

Time of Experiment.	Arsenious Oxide added.	Arsenious Oxide found.	Arsenious Oxide left in Apparatus.
15 minutes	30	10	20
„	20	2	18
„	20	5	15

ARSENATES.

Time of Experiment.	Arsenate added Equivalent to Arsenious Oxide.	Arsenic found.	Arsenic left in Apparatus.
45 minutes ...	30	10	20
	20	1	19
	20	2	18
	20	5	15
	20	10	10
	10	7	3

In the case of the arsenates, it will be seen the electrode had in some of the experiments become as sensitive as cadmium or lead, from which it may be concluded that the supertension of arsenic is large. An endeavour was made to deposit arsenic chemically on copper for the use of electrodes, but in this case a mirror was always obtained in the blank test. The lead, copper, and nickel electrodes were cut from sheets of the pure electrolytic metals. Iron was deposited on a sheet of the same metal, a thickness of at least 1 mm. being necessary, otherwise it was all dissolved during the experiment. The cadmium, tin, and silver were deposited on platinum foil. It may be remarked that it was found almost impossible to get two electrodes to give the same results, although prepared apparently in the same manner. Thus the copper electrodes I. and II., which were cut from the same sheet and cleaned in different ways, gave different results. Further, the introduction of salts of the metal composing the cathode often interfered seriously with the reaction. In the case of arsenate solutions the formation of the mirror was much slower than with arsenites, and for this reason the experiments were continued for three-quarters of an hour in the former case. The results were also invariably lower, which is partly accounted for in the following way: Arsenic pentoxide forms with water a hydrogen salt (acid), which must be partially dissociated in solutions as dilute as those we have used. The ions thus formed must, of course, participate in the conveying of the current from pole to pole, the cathion moving to the negative pole, while the anion (AsO_4) travels to the anode in the opposite direction, and so causes the arsenic to be removed from the sphere of action of the hydrogen. To test this experimentally 0·02 mgr. arsenious oxide was introduced into the cathode compartment, and the apparatus allowed to run for a quarter of an hour. Ten c.c. were then removed from the anode compartment and the arsenic determined in the Marsh apparatus, when a deposit corresponding with 0·005 mgm. was obtained. This experiment was repeated with a copper cathode, using in this case 0·2 mgr. arsenious oxide. A mirror, corresponding to 0·005 mgm., was obtained from the contents of the anode compartment, although the solution contained a little copper sulphate. We may add that we also worked with the apparatus of Sand and Hackford, but, as with our own, the results were too low in the case of arsenates.

In what has gone before we have assumed that the presence of any metal capable of producing insensitiveness in zinc is accompanied by a fall in the potential of the hydrogen formed, as this appeared not only to explain the results we had obtained, but also to be in accord with the published observations of other workers. We felt, however, that the investigation would not be complete without an attempt to verify this suggestion by direct experiment, and we therefore proceeded to determine the potential of dissolving zinc in the presence of various salts. The potentiometer method was the one employed, as it was considered the most suitable for the purpose, and the results are given in tabulated form below. The E.M.F. of the following element was measured: mercury-mercurous sulphate, N sulphuric acid —zinc. For each experiment a new piece of zinc was used, the surface of which was cleaned with strong nitric acid. After having been thoroughly washed with water this was connected with the normal electrode and the potential read off. This varied somewhat and never became quite steady, notwithstanding the fact that the acid around the zinc was well stirred. The salts referred to in column 1 were then added and the sudden changes in potential recorded. These are given in columns 3 and 4, the signs + and – denoting respectively a rise or fall in the E.M.F. of the element in question.

Salt added.	Quantity.	E.M.F. of Element.	Change of E.M.F.	Remarks.
None	—	1·398 volts	—	E.M.F. was falling very slowly, but almost steady.
Cadmium sulphate ...	2·0 grams	1·419 volts	+0·021 volt	Slowly rising.
None	—	1·420 volts	—	E.M.F. dropping slowly.
Cobalt sulphate ...	2·0 grams	1·400 volts	–0·02 volt	E.M.F. dropping. Very small quantity Co deposited.
None	—	1·412 volts	—	Dropping slowly.
Platinum chloride ...	0·2 gram	1·237 volts	–0·175 volt	Dropping rapidly.
None	—	1·398 volts	—	Dropping slowly.
Copper sulphate ...	2·0 grams	1·375 volts	–0·023 volt	Dropping.

In all these experiments 600 c.c. of normal sulphuric acid was used, and the salts added were dissolved in similar acid. The zinc was never completely covered, as the solutions used were very dilute. For this reason stronger solutions were employed in the next series of experiments.

Salt added.	Quantity.	E.M.F. of Element.	Change in E.M.F.	Remarks.
None	—	1·403 volts	—	
Cadmium sulphate ...	20 grams	1·372 „	–0·031 volt	Cadmium sulphate not quite pure
None	—	1·430 volts	—	
Iron sulphate ...	20 grams	1·343 „	–0·087 volt	
None	—	1·416 volts	—	
Cobalt sulphate ...	20 grams	1·320 „	–0·096 volt	
None	—	1·428 volts	—	
Copper sulphate ...	20 grams	1·146 „	–0·282 volt	

At this point 10 grams cadmium sulphate were added, although the solution still contained copper sulphate, and the E.M.F. first rose to 1·30 volts, having increased 0·154 volt, and still continued to rise. In the next experiment the salts were added one after the other without changing the solution.

Salt added.	Quantity.	E.M.F. of Element.	Change in E.M.F.	Remarks.
None	—	1·417 volts	—	
Iron sulphate ...	2·0 grams	1·377 „	- 0·040 volt	
„	2·0 „	1·369 „	- 0·008 „	
Cobalt sulphate ...	2·0 „	1·331 „	- 0 038 „	
Copper sulphate ...	10·0 „	1·219 „	- 0·112 „	
Platinum chloride ...	0·1 gram	1·078 „	- 0·141 „	
			0·339 „	

There can be no doubt that the reducing efficiency of the hydrogen obtained as the result of the interaction of metals and acids, is dependent on a number of factors both chemical and physical, and that even those reactions which are apparently the most simple are in reality exceedingly complex. We think, however, that the experiments, of which an account is given in this paper, show very clearly that among those factors the question of "potential" or "supertension" plays a very prominent and important part.

In conclusion, we desire to record our best thanks to Mr. E. R. Saunders for placing at our disposal the delicate apparatus necessary for the determination of the potential changes above referred to.

DISCUSSION.

The PRESIDENT (Mr. Bevan) having invited discussion,

Professor W. A. TILDEN said that he had come to hear this paper quite in the character of a novice, for, although it was perfectly true that he had occupied himself considerably with the subject three years ago, he had not had any opportunities of pursuing it since, and he should hesitate to express any very strong views in the presence of so many who were accustomed to apply the Marsh-Berzelius method daily in their work. The authors, however, had not only given an exposition of the results of their experiments which had led to the very great improvement in the method which they had described, but had also furnished theoretical views as to the nature of the changes that took place. With regard to those theoretical views, he confessed that he did not feel at that moment as though he could accept them quite without reserve—at any rate, without a little time to think about them. These reducing effects had been spoken of as the effects of the hydrogen, but that was a sort of elliptical phrase which no doubt had a good deal behind it. He was not quite sure what the authors' view was as to the exact action of the hydrogen in the process. At the same time, in every one of these cases there was a couple, and it was the couple that was the active agent. Of course, it was perfectly true that hydrogen, even in the molecular form, had a greater power of reduction than was commonly supposed. He did not know whether everybody was aware that even hydrogen gas was capable of reducing some metals from solution. Solution of silver nitrate, for instance, could be made to deposit metallic silver by merely bubbling hydrogen gas through it. It was probable, however, that the active hydrogen in the case they were considering was not in the molecular form, but was ionized. In the reduction of arsenious acid or of arsenic acid, the process necessarily took place in two stages. First, there was the removal of the oxygen, and there might also be some deposition of elemental arsenic; then came the production of the hydrogen compound. At present he was certainly rather inclined to cling to the idea that the differences which had been observed in the effects of different metals were really not so much attributable to the difference of potential in the couples employed as to the peculiar surface action which many of them were capable of exercising. He could not help thinking that some of them simply adhered to the deposited arsenic in some way which had been spoken of as "surface action," though nobody knew what that meant; and that, in consequence of this surface action, the arsenic was not affected by the reducing action of the hydrogen, and not carried over in the form of a hydride. The reduction of arsenic acid was notoriously difficult, but he confessed that he was a little reluctant to accept the explanation which the authors had given. White arsenic dissolved in water undoubtedly contained an acid—*i.e.*, it contained hydrogen ions and arsenious ions as well as arsenious oxide. Of course, it was perfectly true that the greater part of the arsenious acid was in a state of dissociation, but the comparison which the authors instituted in the two formulæ they had given did not, to his mind, afford a sufficient explanation of the difference. That arsenious oxide was very much more easily reducible under all circumstances than arsenic acid was an elemental fact which did not admit of further analysis. At any rate, if the same experiment were made with arsenious acid, instead of with arsenic acid, he should rather expect to find that the arsenious ion would in like manner find its way by diffusion into the anode chamber. His experience of the practical application of the Marsh-Berzelius process was so limited that he did not think that he could add anything to what the authors had said on this head. He confessed, however, that if he had occasion to use the process he should at once proceed to the use of the electrolytic method, by which, in his experience, using clean platinum electrodes, there was no difficulty in getting the whole of the arsenic out of an arsenious solution in all ordinary cases. What might be the cause of the retention of the arsenic in some cases where there were very complicated mixtures of organic materials was, of course, a matter about which there might be considerable difference of opinion. When it was necessary to use zinc the authors had undoubtedly placed in the hands of the analyst a most valuable modification of the method. It was certainly a most remarkable thing that cadmium and tin stood apart, as they apparently did, from all the other metals which they might be expected to resemble in this respect.

Mr. Herbert Jackson said that he was sure that the authors, to whose paper he had listened with very great interest, would not mind his saying that he did not think their explanation included everything. While the experiments and their numerical results were being detailed, it had passed through his mind that a closer investigation of the arsenide combination was necessary. He believed, however, that the authors' idea with regard to potential was in the main correct, in the following sense: The arsenic remaining in the flask would, if a sufficient time were allowed to elapse, be practically all deposited on the zinc. The potential difference between zinc contaminated with iron and zinc arsenide was probably low. It would be necessary to have a knowledge of the potential difference between pure zinc and arsenic and between zinc and zinc arsenide. Now, that potential difference might be regarded—though it was true that the view was a somewhat mechanical one—as representing the difficulty of breaking up the complex molecules into simpler constituents; and if a larger potential difference were required before there was any change in the molecular complexes of the metals, then he could quite understand that, with a high potential difference, there would be a greater number of arsenic and zinc molecules in a simple and active condition. He had made an attempt to obtain a distinction between the lines in the spectrum of arsenic (in view of the possibility of the separation of two arsenic spectra) by means of some experiments in vacuum tubes with a high-tension current, which might be referred to now because, to his mind, there was no necessary distinction in kind between a spark in a vacuum tube, an electric arc, and a current in an electrolyte. In those experiments, arsenic electrodes having been found inconvenient, he had used electrodes of zinc alloyed with arsenic in an electrolyte of water vapour at a pressure of about 0·5 mm., with a view to obtaining arsenic hydride. The potential difference under those conditions was apparently too low to give more than the spectrum of water vapour. On introducing a spark-gap, however, a certain number of arsenic lines came up, but only those that would be expected—namely, the strong lines of shorter wave-length that were usually the first to appear when any metal was being exposed to electrical oscillation. He had experimented with other alloys, similar in character to those with which the authors had worked, and his observations—in the case of a lead-arsenic alloy especially—bore out what they had said as to the effect of an increase in potential being more completely to separate the arsenic from combination and to convert it into arsenic hydride. He did not think, however, that the full explanation with regard to potential had yet been arrived at, for the number of possible combinations was very large, and the question of the molecular complexity of the arsenides came in as well. He was sorry that the authors had not been able to deal more fully with aluminium. It was a well-known fact that in presence of aluminium in potash solution the arsenic mirror could be obtained from arsenites, but not from arsenates. The potential difference in a zinc-aluminium couple rose very rapidly indeed, and if the surface of aluminium were examined after the rise of potential it would be found to be a very finely divided surface, which, if washed and dried *in vacuo*, and then exposed to the air, oxidized very rapidly, indicating a finely-divided and active state of the aluminium. He was therefore disappointed that the authors had found aluminium unsuitable owing to its yielding such a peculiar substance, and should much like to know what it was. Although aluminium in potash solution would not, under ordinary conditions, give any arsenic hydride with an arsenate, yet with a high-tension current an arsenate could be reduced from such a solution almost as easily as an arsenite. All this went to show very strongly that the question was one of potential difference, and that it was necessary to consider the relation of the arsenic to the other metals, and also of these metals to hydrogen. The influence of cadmium showed that the arsenic would not come off unless the potential was relatively high, and a state of high potential was against the existence of polymeric modifications of the metals on their immediate surfaces. In the case of iron this was well shown by the differing views of Caspari, who gave it, with platinum, a very low value, and of Tafel, who placed it high in the scale. If a low electromotive force were used, the iron acted as stated by Caspari, whereas when a high electromotive force was used, as was the case in Tafel's experiments, the iron probably assumed a passive state. With regard to platinum, he remembered particularly the experiments made by Professor Bloxam on the electrolytic separation of antimony and arsenic, in which the mixed antimony and arsenic sulphides were simply electrolyzed in a porous cell with platinum electrodes in dilute sulphuric acid, when the hydrogen at the cathode reduced the arsenious sulphide and the mirror was obtained perfectly, whereas the sulphide of antimony was left in suspension and did not show. No explanation, however, even if correct, could add to the great practical value of the authors' work on this subject, and the criticisms he had ventured to make, so far from detracting from its value, only showed how important it was from a theoretical point of view also.

Dr. Moritz said that the means which the authors had placed in their hands for dealing with the insensitiveness of zinc were of the greatest value, for insensitive zinc often caused much inconvenience and delay. He should like to ask—though it was perhaps hardly within the scope of the paper—whether the treatment of the zinc with cadmium would have the desired effect on iron and copper introduced in minute quantities in the materials to be analysed, because the products with which he was most concerned did often contain minute quantities of iron and copper.

Mr. Blount said that—viewing the matter, not as one of arsenic determination, but as an electrolytic question—the experts appeared to disagree as to whether the evolution of arsenic depended upon some critical difference of voltage between the metals concerned, or on the absorption, or *ad*-sorption, of the arsenic by the other metals. That might, it seemed to him—he made the suggestion for the authors' consideration—be determined by using electrodes of the metals the behaviour of which it was desired to examine. His point simply was that, if it were a fact that the arsenic came away more readily when the hydrogen was liberated at a particular potential, that potential could be more accurately determined electrolytically by using a suitable electrode of the metal than by adding a salt of that metal to a mass of zinc and so constructing a composite electrode.

Mr. Law, in reply, referring to the reducing action of molecular hydrogen, said that the potential of the platinum black electrode was just slightly higher (namely, 0·005 volt) than the potential of pure hydrogen, so that the reducing action of pure molecular hydrogen might be expected to be almost equal to that of platinum black in the presence of electrolytic hydrogen. There was, according to Caspari, a very marked retaining effect on the hydrogen, due to surface action, and to this retention was due the phenomenon of supertension. The greater the retaining effect on the hydrogen, the higher the supertension was, and consequently the more efficient the electrode was as a reducing agent. Arsenious oxide, as was well known, did not, practically speaking, form any acid. In these very dilute solutions the probability was that any arsenious acid present dissociated first of all into water and

As_2O_3, and that the arsenious oxide itself dissociated further into arsenic and oxygen. As a matter of fact, if an electric current were passed through a very dilute arsenious oxide solution, using a copper electrode, metallic arsenic was actually deposited on the cathode, and did not wander to the anode, as it would if the ion were a complex one. Further, the conductivity of arsenates and arsenites—or rather of arsenic acid and arsenious acid—was entirely different. For instance, in the case of arsenic acid the conductivity was comparatively high. Arsenic acid was a strong acid in comparison with arsenious acid. The latter was a substance which had practically lost all its acid properties, and its conductivity was almost inappreciable. In addition to this, full deposits were obtained from arsenious oxide solutions, which would not have been expected where arsenic formed part of a complex anion. He must confess that this paper did not extend quite as far as Mr. Jackson would like it to go. They did not deal with the exact mechanism of the reaction, but it was quite possible that complex arsenic compounds were actually formed and then reduced. Possibly in a future paper they might be able to do something in that direction, but at present they had not gone so far. It was, however, a fact that as the potential increased more and more arsenic was reduced, and they had laid down this as one of the factors determining the reduction. They did not say that it was the only factor, but they regarded it as an important one. There might, of course, be others. There might, for instance, be complex arsenides formed, but these they had not attempted to deal with. He was not quite sure as to the voltage used by Tafel in his electrolytic work, but Tafel mentioned that a lead electrode gave a voltage of about 4, using a porous pot and a current of 1 ampère. In this paper no details were given of any break or rise.

Mr. Jackson said that the rise was very slow.

Mr. Law said that Tafel had used a current of from 1 to 3 ampères. With respect to small traces of iron, nothing conclusive could be stated in the present paper, but in a future communication full particulars would be given. They had not themselves used electrodes of different metals, but several other investigators had done so, and, in fact, Caspari's table was compiled from results obtained from wires of these pure metals, while numerous other investigators had studied the action of such electrodes. For that reason they had not considered it necessary to use various electrodes in that way. They had, however, used cathodes covered with cadmium, but had not measured the potential.

Dr. H. Sand, who has had the opportunity afforded him of perusing an advanced proof of the paper, has favoured us with the following remarks:

I have been very much interested by Messrs. Chapman and Law's important paper, more especially by the experiments on the sensitization of insensitive zinc in the ordinary Marsh-Berzelius test.

The fact that the authors obtained different results when using an iron electrode from those recorded in Tafel's paper, as well as in that of Mr. Hackford and myself, is, I think, very probably to be ascribed to the circumstance that they experimented with electrolytically-prepared iron, whereas we used thin iron wire such as is employed for the standardization of permanganate solutions. Our experiments (Table IV. of our paper) on solutions containing $FeSO_4$, it will be found, agree with those of

Messrs. Chapman and Law. The difference seems the less surprising as electrolytic iron very probably contains hydrogen, and is known to differ from ordinary iron in other ways, such as in its great resistance to rusting, etc.

The difference between Messrs. Chapman and Law's paper and ours on the behaviour of a lead electrode towards arsenate solutions is not very great. The lead they experimented with appears to have required rather longer than ours for the complete reduction of the arsenate. This being so, it does not seem surprising that as much of the arsenate should have been lost by diffusion, if not through leakage through the partition, as by the cause mentioned by the authors. In any case I think the difference would be more marked when experimenting with large than with small quantities.

With regard to the authors' theoretical introduction, I should be inclined to the view that the hydrogen, before being given off in the form of bubbles, is contained in a state of extreme supersaturation in the layer of solution immediately adjoining the electrode. In this case M_H should not be considered constant, but a function of the supertension in such a manner that the supertension would, according to our present theoretical views, enter into the expression for the equilibrium concentration of the arsenic hydride in an exponential form thus:

$$M_{AsH_3} = K_1 M_{As} . K_2^{P},$$

where P is the supertension and K_1 and K_2 are constants. Personally, however, I think it corresponds best to our actual knowledge of the matter to say that the rate of evolution of arsenic hydride is an unknown function of the supertension and the concentration of the arsenic compounds, this function increasing from zero upwards, as the concentration of the arsenic and the supertension increase from certain definite finite values.

* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Observations on the Analysis of Food Materials. H. Leffmann. (*Chem. Zeit.*, 1905, xxix., 1086.)—I. *Detection of Abrastol* (*Asaprol*).—Abrastol is a β-naphtholsulphonate; it has antiseptic properties and can be used as a food preservative.

The author proposes the following process for its detection in milk: Ten c.c. of milk are treated with 0·5 c.c. of a solution of acid mercuric nitrate (prepared by dissolving mercury in twice its weight of nitric acid and diluting with five times the quantity of water). If abrastol is present a yellow coloration is quickly shown. A control experiment with pure milk should be made at the same time. In wines, etc., abrastol may be detected by acidifying with a few drops of dilute sulphuric acid and shaking with an equal volume of ether, chloroform, etc. The solution is drawn off, and a few drops of the mercuric nitrate solution added and the liquids shaken

Abrastol is indicated by the mercury solution turning yellow, and finally bright red. Benzoates and salicylates give no reaction in this test.

II. *Estimation of Methyl Alcohol in Presence of Formaldehyde.*—The formaldehyde is first removed by addition of potassium cyanide solution; sufficient cyanide has been added when a sample gives no blue colour on adding phenylhydrazine hydrochloride, some very dilute, freshly-prepared nitroprusside solution, and then sodium hydroxide (phenylhydrazine nitroprusside test). The liquid is then distilled, and the methyl alcohol estimated in the distillate by conversion to formaldehyde in the usual way. The method gives much better results than the ammonium hydroxide or hydrogen peroxide processes. E. K. H.

A Volumetric Method of Estimating the Cinchona Alkaloids by means of their Double Thiocyanates. P. W. Robertson. (*Proc. Chem. Soc.*, 1905, xxi., 242.)—Many alkaloids give precipitates with ammonium thiocynate in the presence of a zinc or mercury salt. Other metallic salts act in a similar manner, but zinc forms the most insoluble precipitate. The alkaloids most sensitive to this reaction are quinine and the cinchona alkaloids, 1 part of quinine in 50,000 parts of water giving a distinct turbidity when treated with an excess of zinc sulphate and ammonium thiocyanate. These precipitates prove to be double salts of great complexity, cinchonine ammonium zinc thiocyanate having the formula:

$$4C_{19}H_{22}ON_2.3Zn(CNS)_2.2NH_4CNS.{}_4HCNS.,$$

which closely resembles that of herapathite or iodoquinine sulphate:

$$4C_{20}H_{24}O_2N_2.3H_2SO_4.2HI.4I_2.3Aq.$$

The determination of the amount of thiocyanate removed from solution by the alkaloids is said to form an accurate and rapid volumetric method of determining quinine in drugs and for the assay of crude cinchona bark. W. P. S.

Assay of Calabar Beans. Beckurts. (*Apoth. Zeit.*, xx., 670; through *Pharm. Journ.*, 1905, vol. 75, p. 584.)—Physostigmine can be completely extracted from an aqueous solution by ether, and the following method is proposed for the determination of the ether-soluble alkaloid present in the seed; the extracted alkaloid being subsequently titrated, using iodeosine as indicator. Twenty grams of the seed are shaken for three hours with 120 grams of ether and 10 c.c. of 10 per cent. potassium hydrogen carbonate solution. Ninety grams of the clear ether are then poured off, distilled to one-half, transferred to a separating funnel, and shaken out several times with small quantities of $\frac{N}{10}$ hydrochloric acid, 10 c.c. of petroleum spirit being added to prevent the formation of emulsion. To the united acid extracts 45 grams of ether and 10 c.c. of 10 per cent. potassium hydrogen carbonate are added, and the mixture well shaken. After separation has taken place, 30 grams (equal to 10 grams of seed) of the ether are decanted off; 10 c.c. of $\frac{N}{100}$ hydrochloric acid, 20 c.c. of water, and 5 drops of an alcoholic solution of iodo-eosine are added, and the excess of acid titrated back with $\frac{N}{100}$ sodium hydroxide. The method is also applicable to the extract. The author found 0·0825 per cent. of alkaloid in the seeds, and from 1·25 to 1·3 per cent. in the extract. W. P. S.

Assay of Coca Leaves. Greshoff. (*Apoth. Zeit.*, 1905, xx., 291; through *Pharm. Journ.*, 1905, vol. 75, p. 724.)—The following method is employed at the Haarlem Colonial Museum for determining the value of Java coca leaves: 30·5 grams of the dry, finely-powdered leaves are heated on a water-bath at about 80° C. under a reflux condenser for two hours with 300 c.c. of alcohol, the weight of the flask and contents having been noted. After cooling, the quantity of alcohol lost is made up, and 150 c.c. (equivalent to 15 grams of leaves) are filtered off and evaporated, with constant stirring, nearly to dryness. When cold, the insoluble residue is collected on a filter and washed with tepid water until about 60 c.c. of filtrate have been collected. This filtrate is shaken out twice with 30 c.c. of ether, which are rejected; the aqueous solution is then rendered alkaline with ammonia, and extracted with three successive quantities of 30 c.c. of ether. The united ethereal extracts are evaporated, and the residue dried in a water-oven, a current of dry air being meanwhile drawn through the flask to remove a volatile base with a strong tobacco-like odour. The amorphous straw-yellow residue is then dissolved in a little 1 per cent. sulphuric acid; the acid solution is again made alkaline with ammonia and the extraction with ether repeated. The residue obtained on evaporating the ether is dried for three hours and weighed. The following quantities of alkaloid were obtained by the above process in various samples of leaves: Young leaves, 2·02 per cent.; old leaves, 0·78 per cent.; selected apical leaves, 2·1 per cent.; leaves from the base of the plant, 1·2 per cent. The author considers that commercial samples of the leaves should not contain less than 0·6 per cent. of the alkaloid, calculated on the dry substance.

W. P. S.

The Determination of Iron in Ferrum Redactum. H. Cormimbœuf and L. Grosman. (*Ann. de Chim. anal.*, 1905, vol. 10, p. 420-422.)—The following modification of the iodine method is recommended as simpler and more reliable than that given in the German Pharmacopœia: 1 gram of the reduced iron is treated with 25 c.c. of a double normal solution of iodine (254 grams of iodine, and 360 grams of potassium iodide per litre), and the mixture allowed to stand for at least six hours with occasional agitation, after which a large excess (250 to 300 c.c.) of water is added, and the residual iodine titrated with standard thiosulphate solution. Each c.c. of the iodine solution corresponds to 0·056 gram of iron (*cf.* ANALYST, xxx., 338).

C. A. M.

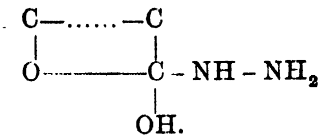
Assay of Sublimate Gauze. Utz. (*Pharm. Post.*, xxxviii., 491; through *Pharm. Journ.*, 1905, vol. 75, p. 691.)—It is well known that in sublimate gauze a part of the mercury enters into combination with the cellulose and cannot be extracted by solvents. The following method, however, originally devised by Rupp, gives excellent results in the determination of mercury in this article: Five grams of the material are placed in a stoppered flask, together with sufficient water to saturate it, and 10 c.c. of a mixture of equal parts of formaldehyde solution and 15 per cent. sodium hydroxide solution are added. After heating for fifteen minutes on a water-bath, the contents of the flask are cooled, 5 c.c. of glacial acetic acid and 5 c.c. of $\frac{N}{10}$ iodine solution are added, and the whole allowed to stand for some time with occasional agitation. The excess of iodine is then titrated back with thiosulphate solution, care being taken to close the flask and shake vigorously towards the end of the reaction; 1 c.c. of $\frac{N}{10}$ iodine indicates 0·01355 gram of mercuric chloride. W. P. S.

ORGANIC ANALYSIS.

The Detection of Formaldehyde. Thévenon. (*Bull. des Sciences Pharm.*; through *Ann. de Chim. anal.*, 1905, vol. 10, p. 433.)—On adding a few crystals of metol (methyl-paramidophenol sulphate) to a solution containing formaldehyde, a garnet-red coloration is produced. Heat accelerates the reaction, but the temperature must not exceed 75° C., or the colour will become yellowish-brown. Acetic or lactic acid or sodium or magnesium sulphate do not interfere with the test, but alkalies change the colour to reddish-brown. The reaction is capable of detecting 1 part of commercial formalin in 10,000. For the detection of formaldehyde in milk, the casein is precipitated by the addition of a few drops of dilute (1 : 5) acetic acid, and the test applied to the filtered serum kept for thirty minutes at a temperature not exceeding 75° C. C. A. M.

Determination of Glycerin according to Shukoff and Schestakoff. (*Zeits. angew. Chem.*, 1905, xviii., 1656.)—From a number of determinations made in the laboratory of the Schlebusch Dynamite Factory, the authors conclude that this method should, for the present, at any rate, be only used as a check on other methods, as, whilst values found by it are generally in good accord with those given by other methods, occasionally it yields results for crude glycerins differing by about 1 per cent. They also state that the extraction with acetone should be continued for six hours, not for four; and they dry their glycerin at 90°, or even 96° to 97° C., instead of at 75° to 80° C., at which last temperature constant weight is obtained only after a very long time. They also omit, as being unnecessary, the initial filtration after adding acid to an alkaline liquid, or alkali to an acid one. A. G. L.

The Identification of Lactones by Means of Hydrazine. E. E. Blaise and A. Luttringer. (*Bull. Soc. Chim.*, 1905, xxxiii., 1095-1104.)—Lactones, if not too impure, can be identified by means of hydrazine, which combines with them to form crystalline compounds with well-marked melting-points. In applying the test the lactone is mixed with an equi-molecular proportion of hydrazine hydrate, and heated on the water-bath until the whole of the water has been expelled. As a rule thirty minutes is sufficient for this when the quantity of lactone does not exceed 2 to 3 grams, but in the case of certain lactones it is necessary to repeat the evaporation once or twice. The dry crystalline product left on cooling the substance is purified by crystallization from absolute ethyl acetate, and its melting-point determined. These compounds are extremely soluble in water and absolute alcohol, but are only slightly soluble in absolute ether. In the author's opinion they are hydrazino-lactones formed by the direct combination of 1 molecule of each constituent, and have the general formula:



Hydrazino-β-γ-dimethylbutyrolactone melts at 96° to 97° C., hydrazino-γ-methyl butyrolactone at 61° to 62° C., and hydrazino-α-methyl-γ-amylbutyrolactone at 116° C. C. A. M.

The Polarimetric Determination of Starch. E. Ewers. (*Zeits. öffentl. Chem.*, 1905, xi., 407-415.)—The starch is brought into solution by successively digesting it with acetic acid, hydrochloric acid, and water, the solution obtained being afterwards clarified and polarized. The details of the method proposed are as follows: 10 grams of the starch or meal are placed in a 200 c.c. flask together with 50 c.c. of glacial acetic acid, and heated in a boiling-water bath for twenty minutes. About 130 c.c. of cold water are then added, and the digestion continued for one hour at a temperature of 45° C., with frequent shaking. After cooling, 2 to 3 c.c. of saturated potassium ferrocyanide solution are added, the volume is made up to 200 c.c., the mixture filtered, and the filtrate polarized in a 200 mm. tube at a temperature of 20° C. The reading obtained gives the amount of soluble carbohydrates, etc., in the sample, and is usually about +0·2° on the Ventzke scale. A second portion of 10 grams of the sample is then digested with 50 c.c. of glacial acetic acid for ten minutes in a boiling-water bath; 10 c.c of dilute hydrochloric acid (1 : 10) are now added, and the heating continued for exactly six minutes. Hot water is then added to make the volume up to 180 c.c., and the mixture heated for a further fifteen minutes. The solution is finally cooled, clarified by the addition of 2 c.c. of the ferrocyanide solution, filtered and polarized as described previously. In the case of potato starch the digestion after diluting with water is omitted. The following readings on the Ventzke scale were obtained, after correction for the soluble carbohydrates, with a number of commercial starches: Wheat starch, +44·7° to +45·4°; rice starch, +45·5°; maize starch, +45·0°; potato starch, +44·5°. The quantity of water, ash, proteids, etc., having been determined in the samples, it is calculated that 10 grams of pure starch dissolved to 200 c.c. and polarized in a 200 mm. tube would give the following readings: Wheat, +52·7° to +52·8°; rice, +52·6°; maize, +52·4°; and potato, +53·3°. W. P. S.

The Determination of Dextrose or Invert Sugar. J. Wolff. (*Ann. de Chim. anal.*, 1905, vol. 10, p. 427-431.)—The method is based on dissolving the reduced cuprous oxide in a solution of ferric sulphate containing sulphuric acid, and titrating the resulting ferrous sulphate with standard potassium permanganate solution. The reagents required are: (1) *Fehling's solution*, consisting of (*a*) 40 grams of copper sulphate per litre, and (*b*) 200 grams of Rochelle salt and 150 grams of sodium hydroxide per litre. (2) *Ferric sulphate solution*—viz., 50 grams of ferric sulphate and 200 grams of sulphuric acid per litre. (3) *Potassium permanganate solution*—5 grams per litre, standardized on $\frac{N}{10}$ oxalic acid solution. In the determination from 5 to 15 c.c. of the 0·5 per cent. sugar solution are treated with 40 c.c. of the mixed Fehling's solution in a conical flask, and the liquid diluted to 60 c.c. and boiled for exactly three minutes, after which it is filtered rapidly through a Soxhlet tube or Gooch's filter. The cuprous oxide is washed by decantation with hot water, and then dissolved in 10 c.c. of the ferric sulphate solution—

$$Cu_2O + Fe_2(SO_4)_3 + H_2SO_4 = 2FeSO_4 + 2CuSO_4 + H_2O.$$

The solution is passed through the filter, the flask and filter washed with an additional 10 c.c. of the ferric sulphate solution, and finally with hot water, and the filtrate and washings titrated with permanganate solution. C. A. M.

New Method of Determining Sugars by Means of the Immersion Refractometer. B. Wagner. (*Zeit. öffentl. Chem.*, 1905, xi., 404-407.)—The amount of cuprous oxide precipitated from Fehling's solution by a sugar may be determined by the immersion refractometer (ANALYST, 1903, xxviii., 91), and the corresponding quantity of sugar thus ascertained. The cuprous oxide is collected in an asbestos filter-tube in the usual way and washed. It is then dissolved in a few cubic centimeters of concentrated nitric acid, and the solution received as it drops from the tube in a small basin, After washing the filter with a little water, the copper solution is evaporated to dryness, the residue is taken up with exactly 5 c.c. of 2 per cent. nitric acid, which has a refraction of 21·6, and then diluted with water to a volume of 10 c.c. The refraction of the solution is now taken. It is necessary to graduate the instrument by taking readings with solutions of copper produced by dissolving the cuprous oxide given by known quantities of sugar, as the refraction is not exactly proportional to the rise in concentration of the sugar solutions 0·002 per cent. of grape-sugar gives a reading of about 19° on the scale of the instrument, whilst 1 per cent. reads 73°. For a difference therefore of 0·998 per cent. of grape-sugar, there is a range of observation of 54°, permitting of considerable accuracy in the the determinations. W. P. S.

The Hydrolysis of Sodium Palmitate. R. Cohn. (*Berichte*, 1905, xxxviii., 3781-3784.)—According to Schwarz (*Zeit. f. öff. Chem.*, 1905, vi., 301) it is impossible to use aqueous acid for the back-titration of the excess of alkali in the determination of acid and saponification values, unless sufficient alcohol be used to prevent the hydrolysis of the soap. It is shown by the author, however, that Schwarz must have failed to observe the neutralization point, for when a solution of palmitic acid in an excess of hot sodium hydroxide solution is titrated with aqueous hydrochloric acid, neutrality is reached when the liquid changes its colour from deep red to faint pink, and with practice the results thus obtained agree within 0·5 per cent. of theoretical amounts. If more acid be then added to the neutralized liquid, the pink colour does not disappear until the whole of the palmitic acid has separated out. It is impossible, with phenol-phthalein as indicator, to obtain quantitative titrations of this hydrolytic dissociation ; but this can be done with methyl-orange as indicator, the reaction then taking place in accordance with the equation :

$$C_{15}H_{31}.COONa + HCl = C_{15}H_{31}.COOH + NaCl.$$

The behaviour of sodium palmitate on titration is thus exactly like that of sodium carbonate, if methyl-orange be used as indicator. The reaction continues alkaline, notwithstanding the addition of acid, so long as free palmitic acid ions from the hydrolysis of the palmitate remain in solution; but as soon as the whole of the palmitic acid has separated out, the free hydrogen ions enter into solution, and the colour of the methyl-orange is immediately changed to red. C. A. M.

Remarks on Lubricants. G. Blars. (*Chem. Zeit. Rep.*, 1905, xxix., 325.)—In a reply to an article by Schreiber, the author declares that in many cases such exacting tests are unnecessary for lubricating oils. The author discusses the flash-point and asphalt content. He proposes for the contract supply of cylinder oils, statements of the specific gravity, and of the viscosity at 150° (after Engler's method). The amount of free acids reckoned as SO_3 must not be more than 0·01 per cent.; free fatty acids must not be formed in a current of steam. A content of asphalt up to 0·4 per cent. only is permissible; cylinder oil must form a clear solution in petroleum benzine of 0·700 specific gravity; for machine oils the requirements must be different. For this case mixtures of mineral and fatty oils give the best results, especially for heavily-loaded axles. The specific gravity must lie between 0·900 and 0·915. The demand for a freezing-point of $-15°$ the author thinks unnecessary, and the flash-point is tolerably beside the question. For dynamo oils, where the axles are not too heavily loaded, the viscosity at 20° must not be more than 15 to 16, and at 50° not more than $3\frac{1}{2}$ to 4.

In a few cases, such as in cart oils and fats, one need only require the lubricant to be free from resin, resin products, and substances increasing the difficulty of working, and have a flash-point not under 100°. E. K. H.

On American Colophony. Paul Levy. (*Zeits. angew. Chem.*, 1905, xviii., 1739.)—From the analysis of a number of salts and esters of abietic acid, the author concludes that the acid has the formula $C_{20}H_{30}O_2$, and not $C_{19}H_{28}O_2$, as given by Mach (*Monatsheft*, 1893, xiv., 186; 1894, xv., 627). A. G. L.

The Determination of Free Hydrocyanic Acid in Aqueous Solution. G. Guérin. (*Journ. Pharm. Chim.*, 1905, xxii., 433.)—The author recommends an addition of borax to the hydrocyanic acid solution prior to the titration with standard silver nitrate or iodine solution.

Modification of Liebig's Method.—Ten c.c. of the hydrocyanic acid solution are mixed with 10 c.c. of a 3 per cent. solution of sodium diborate and titrated with a standard solution of silver nitrate (3·148 grams per litre; 1 c.c. = 0·001HCN), added drop by drop with continual shaking until there is a permanent turbidity. The reactions involved are as follows:

$$(1)\ 2HCN + Na_2B_4O_7 = H_2B_4O_7 + 2NaCN.$$
$$(2)\ 2NaCN + AgNO_3 = AgCN.NaCN + NaNO_3.$$
$$(3)\ AgCN.NaCN + AgNO_3 = 2AgCN + NaNO_3.$$

The solution under examination should be free from ammonium salts, though if necessary they can be converted into borate by the addition of a slight excess of a saturated solution of boric acid.

Modification of the Method of Fordos and Gélis.—Ten c.c. of the hydrocyanic acid solution are mixed with 10 c.c. of the 3 per cent. solution of borax, and titrated with a standard solution of iodine (9·407 grams of iodine, and 16 to 18 grams of potassium iodide per litre; 1 c.c. = 0·001 gram HCN.) until there is a persistent yellow tint.

$$(1)\ 2HCN + 2I_2 = 2CNI + 2HI.$$
$$(2)\ 2HI + Na_2B_4O_7 = H_2B_4O_7 + 2NaI.$$

The benzaldehyde in cherry-laurel water does not interfere with the accuracy of the results. This second method is preferable to the modified Liebig's method for cherry-laurel water and bitter-almond water, since both contain ammonium compounds, which necessitates the use of boric acid if the first method is employed.

C. A. M.

The Detection of Free Hydrochloric Acid in Gastric Juice. Cipollina. (*Riforma Med.*; through *Ann. de Chim. anal.*, 1905, vol. 10, p. 446.)—A reagent consisting of a mixture of aniline water with sodium or calcium hypochlorite has a violet coloration, which is modified by small quantities of hydrochloric acid (above 0·2 per cent.) to a bluish shade, and by still smaller quantities (0·025 to 0·2 per cent.) to violet-red, subsequently changing to yellow. The presence of free lactic acid has no influence upon the colour.

C. A. M.

Analysis of Gambier. Greshoff. (*Pharm. Weekblad.*, xlii., 599; through *Pharm. Journ.*, 1905, vol. 75, p. 657.)—The following method is described for the examination of a gambier, an astringent extracted from a cinchonaceous plant, *Uncaria gambier*.

Water Extract.—Five grams of the powdered gambier are placed in a litre flask which is nearly filled with previously boiled and cooled water; the whole is gently warmed until the gambier is dissolved, and then set aside in the dark for twenty-four hours. After diluting to the mark, the solution is filtered and the extract determined by evaporating 100 c.c. of the filtrate, the residue obtained being dried at 105° C. for three hours.

Tannin.—To 125 c.c. of the solution 2·5 grams of Merck's voluminous alumina are added, the mixture well shaken, allowed to stand for twenty-four hours, then filtered and 100 c.c. of the filtrate evaporated. The difference in the weight of the extract before and after treatment with alumina is taken as tannin.

Crude Catechin.—Two grams of the gambir are powdered with an equal quantity of pumice, transferred to a flask with 50 c.c. of ethyl acetate, and placed aside for twenty-four hours, with occasional shaking. The mixture is then filtered, the residue washed twice with 5 c.c. of ethyl acetate, and the solvent and washings evaporated. The residue is dried at 105° C.

Crystalline Catechin.—The residue of crude catechin is dissolved in 10 c.c. of warm water, filtered, the filter washed with 5 c.c. of water, and the filtrate allowed to crystallize, inoculating if necessary with a crystal of catechin. The crystals obtained are collected on a small filter, washed with 5 c.c. of water, and dried at 105° C. Good gambir should contain from 30 to 35 per cent. of this constituent.

Moisture.—Two grams of the gambier are powdered with 2 grams of dry pumice and the mixture dried at 105° C. The normal amount of moisture in gambier is about 15 per cent.

Ash.—The ash varies from 2 to 4 per cent., and should not exceed 5 per cent.

W. P. S.

The Composition of Certain Marine Algæ (Seaweeds) and of the Products obtained from them. J. König and J. Bettels.

(*Zeit. Untersuch. Nahr. Genusmittel*, 1905, vol. 10, p. 457-473.)—The composition of various Eastern Asiatic seaweeds is shown in the following table, the results expressing percentages on the air-dried substance:

	Water.	Total Proteids.	Soluble Proteids.	Ether Extract.	Nitrogen-free Extract.	Pentosans.	Methyl-pentosans.	Crude Fibre.	Ash.
Porphyra	5·91	34·80	21·94	0·87	47·87	3·37	0·15	2·52	7·51
Porphyra tenera	4·57	34·19	21·75	0·59	46·49	3·79	0·30	2·50	7·57
Gelidium raw	7·36	16·06	7·87	0·98	46·34	2·85	0·71	13·21	12·49
Gelidium bleached	6·82	17·31	7·37	0·73	50·47	3·41	1·13	13·39	5·74
Gelidium cartilagineum	13·00	17·00	7·37	0·80	40·16	3·35	0·91	12·90	11·88
Laminaria	6·16	8·19	5·68	0·50	36·04	6·66	1·12	11·27	30·06
Laminaria japonica	4·20	7·81	5·44	0·39	37·02	8·12	0·84	12·33	29·29
Cystophyllum	16·82	8·12	3·13	0·50	16·35	8·79	1·33	25·91	21·18
Cystophyllum fusiforme ...	15·15	8·06	4·25	0·43	17·43	10·87	1·37	26·16	20·53
Enteromorpha compressa ...	14·17	9·06	5·50	0·20	36·28	7·37	16·52	5·30	12·12
Ecclonia bicyclis	11·56	13·62	7·50	0·28	35·35	5·33	1·06	14·08	18·72
Undaria pinnatifida	9·22	14·00	5·31	0·65	15·12	6·40	0·25	9·23	35·13

Porphyra and gelidium were found to contain *i*-galactose and *d*-galactose, the respective products of these two seaweeds—namely, nori, or vegetable isinglass, and agar-agar, also yielding the same sugars. The quantity of the seaweeds at the author's disposal did not permit of the detection of other hexoses, such as mannose, which Tollens has stated to be present in these algæ. In the case of enteromorpha compressa, however, rhamnose was certainly present. It will be noticed that the specimens containing notable amounts of pentosans also yielded methylpentosans.

The authors have also analysed two samples of edible birds'-nests, with the following percentage results:

Origin.	Water.	Total Proteid.	Fat.	Nitrogen-free Extractives.	Pentosans.	Crude Fibre.	Ash.
East Coast of Java ...	10·42	57·37	0·09	21·98		1·40	8·74
Hongkong	16·33	54·72	—	—	trace	—	5·51

The composition is quite different to that of other seaweed products (agar-agar and nori). Whilst the quantity of proteid is very large, the amount of carbohydrates is small—15 to 20 per cent.—and there is no reason to doubt the conclusion that edible birds'-nests are formed from the vomit of sea-swallows. W. P. S.

A Sensitive Reaction of Formaldehyde and Nitric or Nitrous Compounds with Proteids. E. Voisenet. (*Bull. Soc. Chim.*, 1905, xxxiii., 1198-1214.)—If a pinch of egg albumin be mixed with 2 to 3 c.c. of water and a drop of formaldehyde solution (5 per cent. solution of commercial formalin), and the liquid treated with three times its volume of hydrochloric acid containing a trace of potassium nitrite (acid of specific gravity 1·18 to which has been added 0·5 c.c. of a 3·6 per cent. solution of potassium nitrite per litre), there is an immediate rose coloration which gradually changes to deep violet-blue. The reaction is produced in the cold, but is favoured by heat, a temperature of about 50° C. being the most suitable for the detection of formaldehyde, of which as little as 1 part in 10,000,000 will produce a rose coloration. For fixed quantities of albumin and nitrous acid the intensity of the coloration increases with the amount of formaldehyde added until it reaches a maximum, after which it decreases, until finally the colour disappears when the aldehyde is in large excess. The reaction is equally sensitive for albumin, and is not prevented by the albumin being present in excess. Sulphuric acid behaves like hydrochloric acid in the test, which can be used advantagously for the detection of nitrous compounds in the former. The reaction is to be attributed to the production of small quantities of scatol and indol formed by the action of concentrated acids on proteids, the oxidation of these compounds by the nitrous acid and the condensation of the oxidation products by the formaldehyde with the formation of the colouring-matter. Most proteids give the reaction, keratin, gelatin, and pure peptones being exceptions. The majority of aldehydes are incapable of replacing formaldehyde in the test, but salicylic and other phenolic aldehydes give the same reaction, whilst acrolein and benzaldehyde give deep blue or bluish-green colorations quite distinct from the formaldehyde violet colour. The reaction is not characteristic of nitrous acid, being also produced by other oxidizing agents, such as chlorine, bromine, iodine, hydrogen peroxide, nitric acid, ferric salts, etc. It is particularly sensitive with nitric acid, of which it is capable of detecting 1 part in 20,000,000. The colour is not produced, or is rapidly destroyed, by reducing agents, including formaldahyde in excess. The reaction can be used for the detection or colorimetric determination of formaldehyde in milk, and for the determination of albumin in urine, in which case the flocculent deposit obtained on coagulating the acidified liquid is dried and examined. C. A. M.

Toxicity of Saponin. Bourcet and Chevalier. (*Bull. Sci. Pharm.*, vii., 262; through *Pharm. Journ.*, 1905, vol. 75, p. 691.)—The saponin of commerce is usually not a simple body, but a mixture of acid saponins, which are innocuous, with neutral sapotoxins, which are decidedly poisonous. The latter, when applied externally to the body, produce local anæsthesia and erysipelatous inflammation. Injected into the blood, they are fatal in doses of 0·0002 to 0·0005 gram per kilo of animal, causing paralysis of the central nervous system, and arresting the cardiac and respiratory actions. In less than fatal doses, the gastro-intestinal organs are strongly inflamed, ending nearly always in perforation. When administered internally, the sapotoxins are less toxic, but cause all the symptoms of catarrh of the mucous membrane. The harmless saponins can be separated from the sapotoxins by making the powdered drug into a paste with calcined magnesia, drying the mixture on a water-bath, and extracting the mass with boiling ethyl acetate. The extract is filtered, evaporated to one-half its volume, and mixed with anhydrous ether, when absolutely innocuous saponin is precipitated. The saponins form a series of bodies of varying composition, and split up when hydrolysed into sapogenin and a sugar, which may be galactose, dextrose, or rhamnose. In certain cases crotonic aldehyde is a product of the hydrolysis. The saponins are not dialyzable. W. P. S.

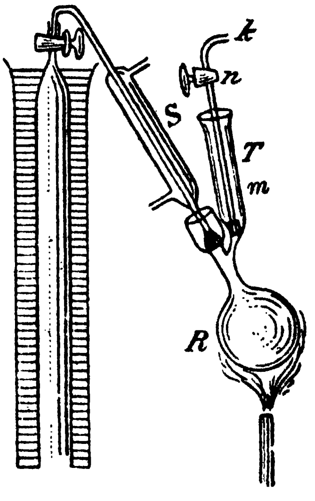
INORGANIC ANALYSIS.

Gasometric Determination of Copper by Means of Hydrazine Salts.

E. Ebler. (*Zeits. anorg. Chem.*, 1905, vol. 47, p. 371.)—Ammoniacal salts of copper are quantitatively reduced by hydrazine salts according to the equation—

$$4Cu(NH_3)_4SO_4 + N_2H_4.H_2SO_4 = 2Cu_2(NH_3)_4SO_4 + N_2 + 2NH_3 + 3(NH_4)_2SO_4,$$

the whole of the nitrogen present in the hydrazine being evolved as gas. The reaction is suited both for the determination of copper and of hydrazine, is carried out in the apparatus, devised by Hempel,* which is shown in the figure set up ready for use. If copper is to be determined, the copper salt is placed in the flask R, together with an excess of aqueous ammonia. The flask and condenser are then exhausted through *k*, attached to a filter-pump; the tap *n* is closed, and a solution of hydrazine sulphate is added from the funnel T until the blue colour is discharged, communication with the flask being established by slightly lifting the tube *k*, the expanded end of which acts as stopper for T. The contents of the flask are boiled until several c.c. of water have been condensed in the burette. It is advisable to use mercury and not water in the burette, as there is a possibility of nitrous oxide being formed instead of nitrogen, although the author has never found this to be the case. The nitrogen is then measured as usual; 1 c.c. at 0° C. and 760 mm. corresponds to 11·31 mgm. of copper. The procedure for determining hydrazine by means of an ammoniacal solution of copper is similar. In both cases the results are good.



If mercury and silver, which interfere with the determination of copper, are present, they may be removed by treating the ammoniacal solution of all three metals with hydrazine sulphate or hydroxylamine chloride; the reduced solution of copper is filtered from the precipitated mercury and silver, evaporated with nitric acid to destroy the excess of precipitant, again evaporated with hydrochloric acid to remove nitric acid, and then treated as above. A. G. L.

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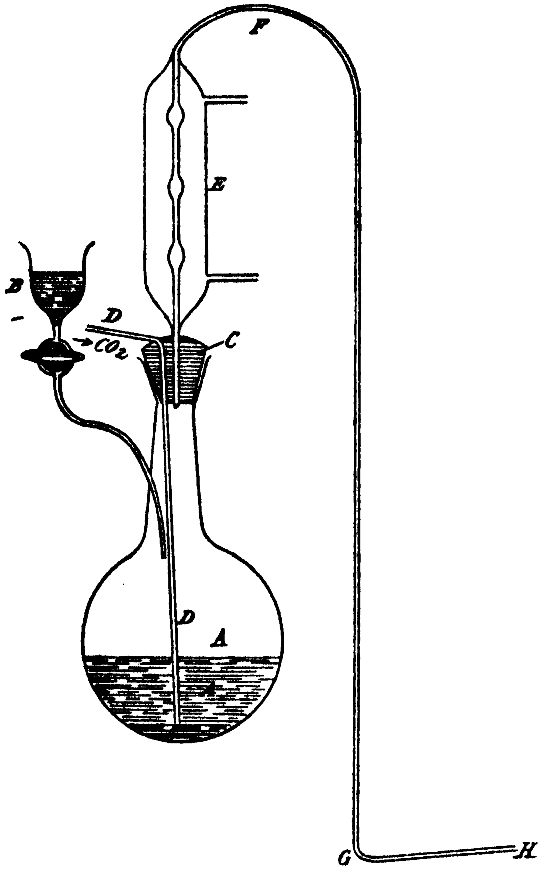
* "Gasanalytische Methode," 3 Auflage, Seit. 62.

The Gasometric and Volumetric Determination of Mercury by Means of Hydrazine Salts, and the Gasometric Determination of Hydrazine by Means of Mercury Salts.



E. Ebler. (*Zeits. anorg. Chem.*, 1905, vol. 47, p. 377.)—The author shows that in aqueous acid or ammoniacal solution mercury salts react with hydrazine salts according to the equation (neglecting ammonium salts)—

$$2HgCl_2 + N_2H_4 = 4HCl + 2Hg + N_2.$$

The reaction is carried out at boiling temperature in the apparatus shown in the figure, the exit tube (H) being connected to a Schiff nitrometer. Either concentrated solutions containing a little hydrochloric acid and an excess of sodium acetate, or else somewhat more dilute ammoniacal solutions, are used. The results, both for mercury and for hydrazine sulphate, are, on the whole, satisfactory.

Another method of determining mercury by means of hydrazine consists in adding a known amount of $\frac{N}{10}$ hydrazine sulphate to the ammoniacal mercury solution, which should contain ammonium chloride, heating on a water-bath until the precipitated mercury has settled, making the whole up to a known volume, and titrating aliquot parts of the filtered solution with $\frac{N}{10}$ iodine, after adding hydrochloric acid and bicarbonate, the reaction between iodine and hydrazine being

$$N_2H_4.H_2SO_4 + 4I = 4HI + H_2SO_4 + N_2.$$

The results obtained in this way are good.

Silver and copper, both of which also reduce hydrazine, may be removed, the former as chloride, and the latter by precipitating the mercury from ammoniacal solution by hydrazine, copper remaining in the solution in the cuprous state. The mercury is then converted into chloride as usual, and this salt is treated as above.

A. G. L.

Further Communication on Simplified Methods of Estimating Potash.

F. Klinkerfues. (*Chem. Zeit.*, 1905, xxix., 1085.)—A fairly lengthy paper, the main points of which only can be given here. The author has given an account of his process (*vide* ANALYST, 1905, xxx., 172). He now makes some further observations on the method. The method does not require the previous removal of sulphates and phosphates. The evaporation with formic acid need not be continued to dryness, but only until reduction of the platinum is complete. Finally, it is unnecessary to dissolve the washed double salt in hot water, but the salt can be washed into the platinum dish and the formic acid added at once. This only requires a small amount of liquid.

The author has also investigated the estimation of nitrogen in potash free ammonium salts by this method; the same degree of exactness is attained as for potash, and the minimum error is not more than 2 mgms. of platinum. This does not depend on the amount of substance taken, but is the unavoidable error of weighing; consequently it is possible, by choosing the quantity of substance used, to make the percentage error as small as may be desired. One gram Pt correspond to 0·1441 grams N.

The author then applied the method to the estimation of potassium in presence of ammonium, substracting the weight of platinum due to the nitrogen (found by an independent process) from the total weight obtained, and thence calculating the potash. The results obtained, however, for potassium ammonium superphosphate were too high, and the author reserves his opinion of the reason for this until he has completed more analyses. Potash free ammonium superphosphate gave exact results.

The author, in the meantime, uses the following process for the mixed superphosphate: The nitrogen is estimated in the ordinary manner, by distilling off the ammonia with potash free magnesia or soda, and the residue in the distillation flask made up to a known quantity; the potash is estimated in a known fraction by the author's process.

Finally, a vacuum dessicator can be dispensed with, as it is immaterial whether the platinum is cooled in vacuo or not. E. K. H.

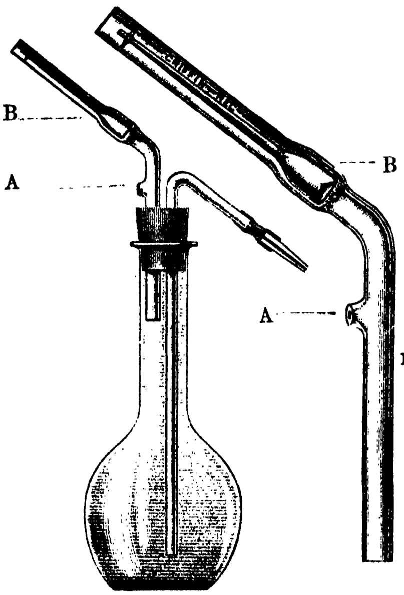
Determination of Sulphur in Pyrites. G. Lunge. (*Zeits. angew. Chem.*, 1905, xviii., 1656.)—Dennstedt and Haszler have recently stated (*Zeits. angew. Chem.*, xviii., 1562) that some basic ferric sulphate may remain undissolved if Lunge's directions relating to the treatment of the residue left on evaporation with "1 c.c. of concentrated acid and 100 c.c. of water" are followed. Lunge now claims that he never meant this to mean that the residue was to be treated with the *mixture* of acid and water, and that he himself always treats first for a short time with 1 c.c. of hydrochloric acid, and only then adds the 100 c.c. of water.

A. G. L.

Estimation of Sulphur in Iron. J. Petrén. (*Chem. Zeit.*, *Rep.*, 1905, xxix., 324.)—The author has investigated the various standard methods. He finds that those methods in which the sulphur is oxidized to sulphuric acid direct, without any separation from iron give the most exact results, but are the most lengthy; those in which sulphuretted hydrogen is evolved are quick and accurate enough for ordinary technical work. E. K. H.

Preparation of Phosphorus Di-iodide. Howard W. Doughty. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1444.)—A flask containing a mixture of 50 grams of iodine and 4 grams of red phosphorus is heated over a naked flame until its contents are thoroughly melted. It is then allowed to cool to 60° C., and 2·5 grams of yellow phosphorus in small pieces are added, when the mass will become solid. The method is stated to be quick and safe. A. G. L.

APPARATUS.



"Continuous - Flow" Wash Bottle.—By means of this arrangement a continuous flow of liquid may be obtained for washing precipitates, etc. In action it is exceedingly simple. The hole A is covered by the thumb, and air blown into the mouth-piece until a certain pressure is induced in the flask. On ceasing to blow, sufficient air is retained by valve B to force a stream of liquid for a short time through the jet. The current stops immediately the thumb is removed from A—in fact, so suddenly that the apparatus may be used for filling small measures, etc., to a definite volume. It is manufactured by Messrs. J. J. Griffin and Co., London.

W. J. S.

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REVIEW.

TECHNICAL METHODS OF ORE ANALYSIS. By ALBERT H. LOW, B.S. New York: Wiley and Sons. London: Chapman and Hall. Pp. 273. Price 12s. 6d.

This work is written for those who are interested in determining only the main constituent in a mineral; consequently the general composition of the ore is dealt with only so far as the means of securing solution may be affected thereby.

After some useful introductory remarks on apparatus, electrolysis, and logarithms, the elements are dealt with in alphabetical order, from aluminium to zinc, each element having, as a rule, a chapter to itself. All the common metals come thus in turn under review, as well as molybdenum, titanium, tungsten, uranium and vanadium, chlorine, phosphorus, silica, and sulphur. Some chapters on the examination of boiler water, coal and coke, and crude petroleum, and, finally, a good index, complete the work.

The methods selected are usually volumetric, and are well suited for technical purposes. A special feature of the work is the fulness of detail with which each step in the method of analysis is described; all the precautions to be observed and pitfalls to be avoided are pointed out. When writing the later chapters the author has resisted the temptation of referring to earlier chapters, in which substantially the same methods of manipulation had been already fully described. By this repetition of details and precautions each chapter is made complete in itself, and the value and convenience of the work is greatly increased thereby.

The printing and general style of the book is good. The alphabetical arrangement of the matter makes it possible to turn at once to what one wants, and the instructions are clear and exact. The work can be confidently recommended.

J. H. B. J.

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HIGH COURT OF JUSTICE.

Size of Sample.

(From the "Pharmaceutical Journal" of December 23, 1905.)

On Thursday, December 20, before the Lord Chief Justice, Mr. Justice Lawrance, and Mr. Justice Ridley, Mary Lowery, of the Stanley Hotel, Hoylake, appealed against a conviction under the Sale of Food and Drugs Acts for selling brandy alleged to be adulterated.

Mr. **Pickford**, for the appellant, contended that the sample taken should be divided into three equal parts, so that each should offer equal facilities for analysis. There was no authority directly in point. He submitted that it was a condition precedent to a prosecution under the Act that the samples taken should be equal.

Mr. **Bankes** said this case was of importance, because in practice it would almost always be absolutely impossible to divide the samples equally as suggested. There was nothing in the section which defined the sizes of the samples to be taken.

The **Lord Chief Justice**, in giving judgment, said the point taken in this case was one of great and general importance—namely, ay or no, was it an objection to a conviction under the section that the samples taken were not equally sufficient for the purpose of enabling a satisfactory analysis to be made. He had come to the conclusion that, although the parts taken need not be exactly equal, at least each must be sufficient for the subsequent purposes contemplated by the Act—namely, that of examination by the public analyst, by any other analyst selected by the person accused, and also by the Government analyst if the justices exercised their power to send it to him under Section 22 of the Act, as they had done in this case. Here they found that the samples were not sufficient, for the public analyst, called as a witness for the prosecution, had stated that he would not be able to get a satisfactory analysis from any sample weighing less than 4 ounces. Therefore the conviction must be quashed, with costs.

Mr. **Justice Lawrance** and Mr. **Justice Ridley** concurred.

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INSTITUTE OF CHEMISTRY DINNER,

DECEMBER 11, 1905.

THE dinner of the Fellows and Associates of the Institute of Chemistry was held at the Whitehall Rooms, Hotel Metropole, on Monday the 11th instant, the President, Mr. David Howard, in the chair.

Owing to the dense fog which prevailed, a number of guests, including Lord Reay, Mr. Justice Buckley, Mr. Justice Swinfen Eady, Professor Meldola (President of the Chemical Society), who had accepted invitations, and Sir R. Douglas Powell, Bart. (President of the Royal College of Physicians), were unable to attend.

Those present included Sir Thomas H. Elliott (Secretary of the Board of Agriculture), Sir Henry W. Primrose (Chairman of the Board of Inland Revenue), Sir Thomas Pittar (Chairman of the Customs Establishment), Sir William Ramsay, Sir Thomas Stevenson, Sir A. R. Binnie (President of the Institution of Civil Engineers), General J. H. Jeffcoat (Master of the Society of Apothecaries), Mr. John Tweedy (President of the Royal College of Surgeons), Mr. J. Gavey (President of the Institution of Electrical Engineers), Dr. Edward Divers (President of the Society of Chemical Industry), Mr. Edward J. Bevan (President of the Society of Public Analysts), Mr. R. A. Robinson (President of the Pharmaceutical Society of Great Britain), Professor W. A. Tilden, Professor J. Millar Thomson, Sir W. H. Bell, Mr. H. H. Cunynghame (Assistant Under-Secretary of State, Home Office), Mr. W. R. Bousfield, K.C., M.P., Mr. J. M. Astbury, K.C.

Mr. R. A. ROBINSON, in proposing "The Institute of Chemistry of Great Britain and Ireland," congratulated the Institute on having attained its present important position. It had nearly 1,200 Fellows and Associates, and was doing an important public work in carrying out the objects of its Charter—namely, "to promote the better education of persons desirous of becoming public and technical analysts and chemical advisers on scientific subjects; to examine candidates and to grant certificates of competency; and to elevate the profession of consulting and analytical chemistry by setting up a high standard of scientific and practical proficiency, and by insisting on the observance of strict rules in regard to professional conduct." As an instance of the success it had attained in promoting these objects, he mentioned that 93 per cent. of the Public Analysts under the Sale of Food and Drugs Acts were Fellows of the Institute.

The PRESIDENT, in the course of his reply, said that the examinations of the Institute were attended now by over one hundred candidates yearly, and arrangements were being made for the conduct of examinations in the colonies. He also alluded to the new scheme of examinations in technical chemistry which the Institute will put into operation next year.

Sir THOMAS STEVENSON proposed the toast of "The Learned Societies and Institutions," which was acknowledged by Mr. Tweedy and Sir A. R. Binnie.

Sir WILLIAM RAMSAY proposed "The Guests," Sir THOMAS H. ELLIOTT and Mr. ASTBURY responded, and the proceedings terminated.

FEBRUARY, 1906. Vol. XXXI., No. 359.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON THE REMOVAL OF ARSENIC FROM HYDROCHLORIC ACID FOR USE IN THE MARSH-BERZELIUS METHOD.

BY ARTHUR R. LING, F.I.C., AND THEODORE RENDLE.

(*Read at the Meeting, December* 6, 1905.)

IT is a well-known fact that hydrochloric acid sufficiently free from arsenic for use in the Marsh-Berzelius method cannot be purchased, nor is this surprising, seeing that the acid acts on the glass bottle in which it is stored, and glass usually contains arsenic. Even the purest hydrochloric acid obtainable in commerce has therefore to be submitted to some preliminary purification before it can be used for any method of determining arsenic.

The Joint Committee of the Society of Chemical Industry and of the Society of Public Analysts recommended a method for the purification of hydrochloric acid which depends in principle on the fact first pointed out by Rohmer (*Ber.*, 1901, xxxiv., 31), that the presence of hydrogen bromide expedites the volatilization of arsenious oxide from a solution when it is distilled in a current of hydrogen chloride and sulphur dioxide. The Joint Committee's method consists in diluting the hydrochloric acid to be purified with water until it has a specific gravity of 1·10, adding about 5 c.c. per litre of bromine, and decolorizing the yellow solution with an excess of sulphurous acid. The acid, after standing for at least twelve hours, is boiled or distilled. The whole of the arsenic is contained in the first portion of the acid (one-fifth of the whole) which passes over.

One of us employed this method regularly until about two years ago, when it was discarded in favour of distillation with metallic copper (the Reinsch method). The reasons for rejecting the Joint Committee's method were threefold. In the first place, it was found very difficult to employ the method successfully with commercial hydrochloric acid. The Committee's instructions are that acid purchased as "pure" is to be treated. In the second place, the yield of arsenic-free acid seldom, in practice, exceeds three-fifths of that originally taken; and, lastly, the method is a lengthy one. The Reinsch method was carried out under conditions devised by Dr. L. T. Thorne, who very kindly gave us complete details of his *modus operandi*. In our experience, Dr. Thorne's method is superior to that of the Joint Committee, as it can be used for the purification of commercial hydrochloric acid. When, however, the acid contains much arsenic the method is extremely slow.

The method of separation of arsenic from non-volatile substances by distillation in presence of hydrochloric acid was considerably simplified and rendered much more exact by Emil Fischer's proposal (*Ber.*, 1880, xiii., 1778) to add ferrous chloride as a reducing agent before distillation. When, however, the residue as well as the distillate has to be examined, the addition of iron is obviously undesirable. In view of this, various other reducing agents have from time to time been proposed, and C. Friedheim and P. Michaelis (*Ber.*, 1895, xxviii., 1414) proposed methyl alcohol for this purpose.

A few months ago H. Cantoni and J. Chautems (*Arch. Sc. phys. nat. Geneve* (4), xix., 364) stated that when a current of air is passed through a solution of arsenious oxide in hydrochloric acid to which methyl alcohol has been added at the ordinary temperature the arsenic is volatilized, probably in the form of the ester methyl arsenite. As soon as this work became known to us we made numerous attempts to purify arsenical hydrochloric acid by making use of the principle just stated, but without success. It was found impossible to remove arsenic by this means at the ordinary temperature.

We need not enter further into a description of the numerous preliminary experiments we carried out before finally adopting our present process. Briefly, the process consists in digesting a mixture of constant-boiling hydrochloric acid and methyl alcohol under diminished pressure in a flask furnished with a reflux condenser for some hours in presence of bright electrolytic copper free from arsenic, and then distilling the acid over similar copper. Digesting with methyl alcohol alone has been found to be capable of removing all but the last traces of arsenic, but the purification is rendered complete and the process considerably expedited by the conjoint use of metallic copper. It is our practice, before commencing the digestion, to add a trace of metallic zinc, and our experience has shown this to be an advantage.

The details of the process are as follows:

To 1,500 c.c. of commercial hydrochloric acid, slightly above 1·1 specific gravity, about 40 c.c. of redistilled commercial wood-spirit are added. The mixture is contained in a Wurtz flask of 2 litres capacity. About 5 to 10 grams of arsenic-free granulated zinc are then added. The flask is connected with the reflux condenser by an ordinary cork in which is fixed a glass rod supporting a coil of electrolytic copper foil, having a surface of about 120 square inches. The side tube of the Wurtz flask having been plugged, the condenser is connected with an exhaust-pump, and the boiling commenced. With acid containing from ½ to 1 grain per gallon, about three hours' gentle digestion is sufficient, the copper being withdrawn and cleaned at least once during this period. During the digestion a black, tarry, fuming liquid distils over, and the greater part of this is caught in a vessel intermediate between the condenser and the pump. It is also advisable to interpose a second somewhat larger vessel containing water to absorb any hydrogen chloride which passes off. This water should be changed as often as necessary.

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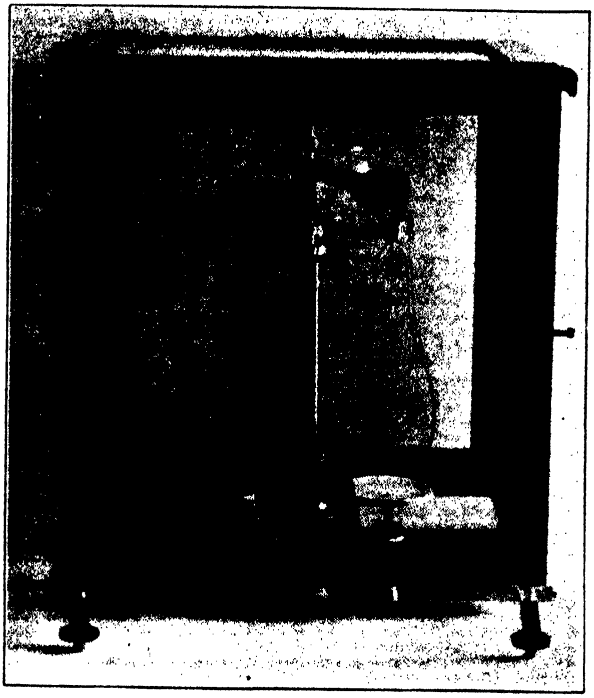
A SIMPLE ARRANGEMENT OF LENSES FOR READING THE GRADUATIONS OF CHEMICAL AND ASSAY BALANCES.

By G. T. Holloway, F.I.C.

(*Shown at the Meeting, December* 6, 1905.)

The usual telescopic or portable lens arrangements are, as shown in the figure, replaced by lenses cemented on the rising glass-front of the balance. Plano-convex lenses cemented with seccotine on the inside of the glass are preferred, but bi-convex lenses cemented at the edges in two or three places to small discs of cork, or other soft material cemented on the glass, may be used, as shown in the case of the upper lens in the accompanying figure.

A 3-inch lens for the pointer reading is a convenient size, but a 2-inch lens is sufficient for reading the beam graduations. A slight movement of the head suffices to bring any part of the beam into the field.



The focal length of the lenses is of little importance, but they must not be powerful enough to produce distortion, or thick enough to prevent the rise and fall of the balance-front on which they are fixed. There is always sufficient space for any ordinary lens, and as the lenses are fixed inside, they remain clean and require no attention or adjustment. As the lenses are not required except when the rider is being used, they are out of the way while the balance-front is raised.

This arrangement is simple and cheap, and absolutely effective in saving strain to the eye, and may be applied with advantage even to a rough balance.

THE FACING OF RICE.

By Cecil H. Cribb, B.Sc. Lond., F.I.C., and P. A. Ellis Richards, F.I.C.

(Read at the Meeting, February 7, 1906.)

It has been a matter of more or less general knowledge for some years that rice is faced, *i.e.*, polished, with the aid of some extraneous substances, but we are not aware of any references to the subject beyond a note in the Analyst (vol. ii., p. 152) on "waxed rice," a statement in the *British Food Journal* (1900, p. 46) that rice is finished with the aid of oil, paraffin oil being sometimes employed, and a paper by Matthes and Müller (*Zeit. öffentl. Chem.*, 1905), abstracted in the Analyst (vol. xxx., p. 206). These authors mention the use of "talc" as a coating material for various cereals, including rice.

It was formerly the custom in some retail businesses, and may be so now, to polish the rice by shaking in a bag of sheepskin or other suitable material, the process being known as "trouncing." With the advent of machinery this simple method is no doubt a thing of the past; but most of the earlier recorded analyses plainly show that, whatever the process employed, it did not as a rule involve the addition of any appreciable amount of *mineral* matter.

The following figures for the total ash of rice are given by the authors named:

	Total Ash.
Boussingault ...	0·5 per cent.
Fresenius ...	0·52
Polson ...	0·9
Poggiale ...	0·32
Cooley's "Encyclopædia" ...	0·5
Jago ("Chemistry of Wheat Flour," p. 233) ...	0·28
Bell ("Analysis and Adulteration of Foods," p. 86) ...	0·28
Wynter Blyth ("Foods: Composition and Analysis," p. 214) ...	0·45
Leffmann and Beam ("Food Analysis," p. 101) ...	0·3 to 0·6 per cent.
Leach ("Food Inspection and Analysis," p. 213):	
Unhulled ...	4·09 per cent.
Unpolished ...	1·15 ,,
Polished ...	0·46 ,,

Hassall, in a complete analysis of the ash, gives the silica as 3·35 and Leach as 6·14 per cent. of the total ash.

We have recently had occasion to examine a number of samples submitted to us under the provisions of the Sale of Food and Drugs Acts, and the very high proportion of ash in some of the samples, together with the presence of a substantial amount of mineral matter insoluble in hydrochloric acid, plainly indicated that some inorganic substance foreign to rice was present.

Table I. gives the total ash, together with the soluble ash and the ash insoluble in hydrochloric acid, in the samples referred to, and it will be seen at a glance that in every case but one the polished samples exhibit an increased total ash and a largely increased insoluble ash, as compared with the unpolished samples, thus clearly establishing the connection between the polishing and the excess of mineral matter

Also it may be mentioned that the sophisticated samples can be at once distinguished by the appearance of the ash as obtained in a muffle furnace. Genuine rice yields a fused glassy mass, whilst the ash of the polished samples exhibits a powdery appearance, and frequently retains, in skeleton form, the shape of the original granules. Further, it will be noticed that the variations in the total ash are almost entirely due to the substances insoluble in acid, the soluble ash being not only fairly constant in amount, but also almost always under 0·5 per cent.—*i.e.*, within the limits of variation of the ash of natural unpolished rice.

TABLE I.

Samples of Unknown Origin.

	Price per lb.	Finish.	Total Ash.	Insoluble Ash.	Residual Ash.
1.	—	Dull	0·29	trace	0·29
2.	1½d.	,,	0·31	,,	0·31
3.	1½d.	,,	0·32	,,	0·32
4.	3d.	,,	0·32	0·005	0·315
5.	—	,,	0·32	trace	0·32
6.	—	,,	0·32	0·01	0·31
7.	—	,,	0·33	0·01	0·32
8.	—	,,	0·37	0·035	0·335
9.	—	,,	0·38	0·03	0·35
10.	—	,,	0·42	0·02	0·40
11.	—	,,	0·57	0·02	0·55
12.	2d.	Polished	0·52	0·18	0·34
13.	—	,,	0·64	0·25	0·39
14.	—	,,	0·70	0·30	0·40
15.	2d.	,,	0·80	0·40	0·40
16.	1d.	,,	0·83	0·41	0·42
17.	2d.	,,	0·83	0·40	0·43
18.	1d.	,,	0·85	0·44	0·41
19.	—	,,	0·85	0·39	0·46
20.	2d.	,,	1·00	0·48	0·52
21.	2d.	,,	1·02	0·53	0·49
22.	1½d.	,,	1·07	0·71	0·36
23.	3d.	,,	1·07	0·66	0·41
24.	2½d.	,,	1·50	1·10	0·40
25.	1½d.	,,	1·60	1·10	0·50
26.	2d.	,,	1·92	1·46	0·46
27.	2½d.	,,	2·00	1·55	0·45
28.	2d.	,,	2·22	1·65	0·57

On further examination it was found that by agitating the rice with water and pouring off the turbid liquid, after the grains had settled to the bottom, and repeating the operation two or three times, the fine particles thus removed from the surface contained practically the whole of the insoluble ash, while the residual grains had the ash of genuine unpolished samples.

TABLE II.

	Insoluble Ash.	Ash of Powder removed by Levigation.
No. 25	1·10	1·05
Patna	0·73	0·68
No. 12	0·18	0·25
No. 26	1·46	1·52

More detailed analyses of the mineral matter in the samples will be found in Table V.

It appears that, while the ash of the unpolished rice contains only a trace of insoluble matter, and from 9 to 11 per cent. of magnesia (MgO) in a soluble form, in the polished rice the insoluble portion almost invariably forms at least 40 per cent. of the total ash, and consists mainly of magnesia and silica.

Although the substance used in the polishing process apparently adds comparatively little to the amount of soluble ash, nevertheless, the latter invariably contains an increased amount of magnesium, the result, no doubt, of the partial decomposition of the silicate by fusion with the alkaline salts of the soluble ash, as the following figures show :

TABLE III.

Rice.	Percentage of MgO in portion of Ash Soluble in HCl.
Carolina, unpolished	11·3 per cent.
„ polished	24·5 „
Patna, polished	23·5 „
Java, polished (A)	20·2 „
„ „ (B)	21·2 „

It may be taken for granted, therefore, that a compound of magnesia and silica is largely, though not universally employed in the polishing process. To get further information we have examined a number of samples of known origin and of every price and quality, very kindly supplied to us by some of the largest wholesale and retail houses in the trade, and the analyses of these are set forth in Table IV., p. 43.

The conclusions already arrived at are fully borne out by the foregoing figures, for with the exception of the samples described as "oil polished," all the polished samples contain excess of mineral matter, showing itself almost exclusively in the insoluble ash.

The absolute identification of the foreign substance is difficult, if not impossible. The powder washed off from the outside of the grains, when examined under the microscope, appears as transparent angular particles of irregular outline, which exhibit no particular characteristics, although they have a general resemblance to mica and talc.

TABLE IV.

Samples of Known Origin.

Rice.	Price.	Finish.	Total Ash.	Insoluble Ash.	Residual Ash.
Carolina	6d.	Dull	0·22	0·01	0·21
„	4½d.	„	0·29	0·02	0·27
„	4d.	Polished	0·68	0·44	0·24
„	4d.	„	0·86	0·41	0·45
Patna:					
Foreign cleaned	3d. to 4d.	„	0·71	0·30	0·41
„ „	2½d.	„	1·07	0·70	0·37
„ „	2½d.	„	1·14	0·73	0·41
Broken, London milled	2½d.	„	0·90	0·50	0·40
„ „ „	2½d.	Slightly polished	0·80	0·42	0·38
English cleaned	3d. to 4d.	„ „	0·87	0·47	0·40
„ „	3d.	„ „	0·92	0·56	0·36
„ „	3d.	„ „	1·08	0·78	0·30
Japan	3d.	Dull	0·22	0·001	0·219
Siam, Dutch cleaned	—	„	0·40	0·03	0·37
Bassein:					
German cleaned	2d.	Polished	0·77	0·35	0·42
„ „	2d.	Polished (contains damaged grains)	1·10	0·60	0·50
Java:					
Natural cleaned	2½d.	Dull	0·33	0·015	0·315
Foreign cleaned	—	Polished	0·78	0·34	0·44
Indigo treated	4d.	„	0·82	0·44	0·38
„ „	2d.	„	1·26	0·90	0·36
English cleaned	2½d.	„	0·71	0·31	0·40
„ „	2½d.	„	0·74	0·43	0·31
„ „	2½d.	„	0·99	0·71	0·28
„ „	2½d.	„	1·14	0·73	0·41
Dutch cleaned	3d.	„	0·40	0·07	0·33*
„ „	3d.	„	0·51	0·01	0·50
„ „	3d.	„	0·54	0·25	0·29
„ „	3d.	„	0·73	0·35	0·38
„ „	3d.	„	0·99	0·49	0·50
Burma	1½d.	Dull	0·30	trace	—
Rangoon:					
Rice with hull	—	Brown	2·62	1·42†	1·20
Rice de-hulled in the laboratory	—	Very dull	1·32	0·13†	1·19
Oil-dressed, in Holland	—	Slightly polished	0·30	0·01	0·29
Broken, London milled	—	Dull	0·52	0·17	0·37
Antwerp milled	—	Polished	1·15	0·82	0·35

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* Probably polished by oil or some other process.

† Practically all silica.

Even the wholesale dealers appear to have no certain knowledge of the *modus operandi* of the polishing process, which seems to be a carefully-guarded trade secret, and no doubt differs slightly in different countries as regards both the method and the nature of the added substances. Steatite, French chalk, powdered talc, and mica are amongst the substances hinted at. We were informed that two of the Java samples had been treated with indigo in order to improve the colour, but we have so far been unable to verify the statement. The following table shows the composition of these, together with that of the insoluble matter actually found in the samples examined by ourselves:

TABLE V.

Polished Rice.	Percentage Composition of Silicious Matter.		
	SiO_2.	MgO.	$Fe_2O_3 + Al_2O_3$
Carolina	61	22	present*
Patna	56	22	5·8
Java	58	24	present*
A. Unknown origin ...	62	22	„
B. „ „	62	25	6·1
C. „ „	62	25	present*
D. „ „	58	28	7·01
E. „ „	61	20	9·00
French chalk	61	29	4·02
Talc	57	30	3·6
Steatite	62	33	3 to 4

Assuming that one or other of these substances is used, the difficult question which always confronts the Public Analyst in such matters at once arises—Is this method of treating rice to be regarded as adulteration?

On the one hand, it must be admitted that it is absolutely unnecessary, inasmuch as there are other modes of polishing which are not open to the same objection; and, moreover, there is no reason whatever, apart from the whims of certain purchasers, why the grains should not be left with their natural surface, as, indeed, is the practice of some of the chief rice-eating nations, notably Japan.

The proportion of foreign matter present in some of the samples is quite sufficient to constitute a fraud, inappreciable, perhaps, by the individual purchaser, but ample to give to one unscrupulous vendor an unfair advantage over his more honest rivals.

Further, on the medical aspect of the case, it has been suggested to us that there is the possibility that even very small quantities of an insoluble mineral substance may be injurious to health, not, of course, owing to any chemical action, but merely by the mechanical irritation it might set up, and because of the possible formation of fæcal concretions.

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* Quantity not estimated.

On the other side, it has been urged that the proportion of foreign matter is at the worst small and at the best negligible; that the amount of irritation likely to be set up must be extremely slight, and would be quite as likely to be beneficial as otherwise by the promotion of peristalsis. As the alleged adulterant is more expensive than the rice itself, there could be no possible temptation to the manufacturer to adopt the process for the purpose of fraud, and in addition to this, it might also be urged that the foreign matter generally is, and always might be, largely removed during cooking. We understand this is the custom of native cooks in India, by whom the rice is always soaked and washed before use.

Whatever may be the opinion in regard to the legal aspect of the question, it is somewhat startling to reflect that an article of diet of such common and widespread use, hitherto accepted as one of the purest forms of vegetable food, should so frequently contain a substance of a foreign nature.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The "Aldehyde" Value of Milk. R. Steinegger. (*Zeit. Unters. Nahr. Genussm.*, 1905, x., 659-671.)—The addition of formaldehyde to milk causes an increase in the acidity of the latter, apart from that due to the acidity of the formaldehyde solution itself. The acidity increases with the quantity of formaldehyde added until it reaches a maximum with the addition of 1·8 per cent., or about 5 per cent. of the ordinary 40 per cent. formalin solution. The difference between the original acidity of the milk (expressed in Soxhlet-Henkel degrees) and the acidity of the same after the addition of 5 or 6 c.c. of formalin is termed by the author the "aldehyde" value of the sample. This value for normal milks varies between 5·8 and 8·5. For the milk from single cows the value remains practically constant. In the case of colostrum the value rises as high as 17·3, but decreases as the milk becomes normal. The increase is observed with boiled milk, showing that the phenomenon is not due to the action of an oxidizing ferment. As the aldehyde value varies with the amount of casein and other proteids in the milk, the increase of acidity is undoubtedly caused by the chemical reaction between the formaldehyde and these proteids. The determination of the aldehyde value is proposed as a means of detecting the presence of added water in milk, as this form of adulteration decreases the percentage of proteids in the milk, and, consequently, the aldehyde value. Abstraction of fat does not influence the value. W. P. S.

The Quantity of Water in Canadian "Domestic" Butter. A. McGill. (*Canadian Inland Revenue Department*, Bull. 107, 1-12.)—By domestic butter is meant butter as found in the retail market, and intended for home consumption.

Of 180 samples of this class of butter examined, only 1 contained an amount of water in excess of 16 per cent. Eight of the samples contained from 14 to 16 per cent., and in 144 samples the water was below 10 per cent. One of the samples contained borate preservative.
W. P. S.

Detection of Phytosterol in Lard in the Presence of Small Quantities of Paraffin, and the Determination of Small Amounts of Paraffin in Lard.

E. Polenske. (*Arbeit. Kaiserl. Gesundheitsamte*, 1905, vol. 22, pp. 576-583.)—The sensitiveness of the phytosterol acetate test (ANALYST, 1902, xxvii., 94) is sometimes vitiated by the presence of traces of paraffin in the fat under examination. The paraffin becomes more and more concentrated as the acetate is recrystallized, until at last the melting-point obtained falls to that of the paraffin—that is, about 53° C. By taking advantage of the slight solubility of phytosterol in petroleum spirit, it is possible to remove the paraffin. The unsaponifiable residue obtained from 100 grams of fat in the usual manner is placed in a cylinder of about 8-c.c. capacity, and treated with 1 c.c. of petroleum spirit (boiling-point below 50° C.). After stirring up the residue with a glass rod, the cylinder is stoppered and placed aside for about twenty minutes. The whole is then poured on to a small pellet of cotton-wool placed in the stem of a funnel, and the residue and filter washed five times with petroleum spirit, using 0·5 c.c. of the latter each time. The residue of phytosterol is finally dissolved off the cotton-wool with a little ether, evaporated, and acetylated. Besides removing the paraffin, the above process concentrates the phytosterol in the residue, as cholesterol is considerably more soluble in petroleum spirit.

For the determination of small quantities of paraffin in fats the crude, unsaponifiable residue from 100 grams of the fat is heated in a strong glass tube with 5 c.c. of concentrated sulphuric acid to a temperature of 105° C. for one hour (a mixture of 40 parts of glycerol and 60 parts of water boils at 104° to 105° C.). After cooling, the contents of the tube are shaken out three times with petroleum spirit, using 10 c.c. each time, and continuing the shaking for one minute. The extracts are united, washed a few times with a little water, the petroleum spirit is evaporated, and the residue weighed after being dried at 100° C. Should only traces of paraffin be present it is advisable to subject the residue to a second treatment with sulphuric acid.
W. P. S.

The Determination of Tannin in Wine.

L. Kràmszky. (*Zeit. anal. Chem.*, 1905, vol. 44, pp. 756-765.)—It is shown that the method of Neubauer and Löwenthal gives not only the amount of tannin, but the sum of the tannin and colouring matters. Barth's colorimetric method has the same drawback, and is also not generally applicable. An ammoniacal solution of zinc sulphate (or of nickel sulphate) yields quantitative results with solutions of pure tannin, which agree well with those obtained by the Neubauer-Löwenthal method, and at the same time does not precipitate the colouring matter of wine. The reagent is prepared by dissolving about 25 grams of pure zinc sulphate in water, adding ammonia until the precipitate first formed redissolves, then an additional 300 c.c. of ammonia, and making up the liquid to a litre. For the determination of the tannin 50 c.c. of a red wine or 100 c.c. of a white wine are rendered alkaline with ammonium hydroxide and heated, no notice being taken of any precipitate that may be formed. The liquid is then treated with 20 c.c. of the reagent and stirred until the zinc tannate separates, after which 300 c.c. of hot water are added, and the whole allowed to stand. The precipitate is repeatedly washed by decantation with hot, slightly ammoniacal water, and finally brought on to the filter (preferably a Gooch's crucible containing asbestos), dried at 100° to 130° C., and weighed. It is then oxidized by means of a few drops of strong nitric acid, and ignited after evaporation of the acid. The difference between the two weights gives the amount of tannin. The author gives a series of results thus obtained with solutions of pure tannin and with wines, including some to which had been added known quantities of tannin. Comparative determinations were made by the method of Neubauer and Löwenthal, the difference between the results in the case of red wines ranging from 0·02 to 0·08 gram. According to Neubauer, a deduction of 0·03 to 0·04 gram must be made for the colouring matters in dark-red wines when the permanganate method is used. As regards the question of gallic acid, the author considers that it is probably only present in traces in sound red wines, but that unsound wines may possibly contain considerably more. Experiments to determine the influence of gallic acid on the determination of the tannin by the zinc method have given the following results: (1) Gallic acid is not precipitated by ammoniacal zinc sulphate solution. (2) Wines to which a known quantity of gallic acid has been added give, on analysis by the method of Neubauer and Löwenthal, a higher value than corresponds to the sum of the tannin, colouring matter, and added gallic acid. (3) In certain cases the presence of gallic acid prevents the precipitation of part of the tannin by ammoniacal zinc sulphate solution. C. A. M.

A Honey Substitute. G. Reiss. (*Arbeit. Kaiserl. Gesundheitsamte*, 1905, vol. 22, pp. 666-668.)—Under the name "Fruktin" an article has recently been placed on sale as a honey substitute. It consists of cane-sugar and a small quantity of tartaric acid, a little caramel being also present. According to the directions given with each packet of the article, 500 grams of the latter are heated to boiling with 150 grams of water, and the mixture then kept in a warm place for half an hour in order to obtain a honey-like product. The author has himself prepared artificial honey by treating "Fruktin" in this way, and from it obtained the following analytical data: Specific gravity of a solution of 1 part in 2 parts of water, 1·11; invert sugar, 33·39 per cent.; cane-sugar, 41·63 per cent.; ash, 0·024 per cent.; polarization of a 10 per cent. solution observed in a 200-mm. tube, +4°35′; after inversion, −2°44′. The proportion of cane-sugar is, as will be seen from the above figures, about four times greater than is found in natural honey, whilst the percentage of ash is much lower. At the same time, a mixture of 1 part of "Fruktin" honey with 3 parts of pure honey would contain about 10 per cent. of cane-sugar, and such mixtures would only differ from natural honey in their low percentage of ash and in the presence of tartaric acid in them. W. P. S.

Valuation of Saffron. A. Jonscher. (*Zeit. öffentl. Chem.*, 1905, xi., 444-447.)—The stigma of saffron possesses a considerably higher aromatic or spice value than the pistil, as it contains more ethereal oil and crocin. The stigma is also superior to the pistil as regards colouring power. An approximate method for determining the value of a sample of saffron consists in boiling 0·1 gram of the air-dried sample with 10 c.c. of 50 per cent. alcohol, at once cooling the mixture, and then allowing it to stand for one hour. The solution is then filtered, and 5 c.c. of the filtrate diluted to 100 c.c. with water. The coloration is then compared with that produced by a good specimen of saffron under similar treatment. W. P. S.

Determination of Vanillin. J. Hanus. (*Zeit. Unters. Nahr. Genussm.*, 1905, vol. 10, pp. 585-591.)—Vanillin is quantitatively precipitated from its aqueous solution by *m*-nitrobenzhydrazide. The presence of the ordinary adulterants of vanillin, such as acetanilide, benzoic acid, salicylic acid, and sugars, is without influence on the precipitation. For the determination of vanillin in vanilla the following method, based on the above-mentioned fact, is described: About 3 grams of the vanilla, in small pieces, are extracted for three hours with ether. The ethereal solution is evaporated at a temperature of 60° C., the residue is dissolved in a little ether and filtered into a small flask, the filter being washed with ether. The ether is again evaporated, the residue heated with 50 c.c. of water for thirty minutes on the water-bath, and the emulsion so obtained precipitated with a solution of 0·2 gram of *m*- nitrobenzhydrazide in 10 c.c. of hot water. The flask and its contents are placed on the water-bath for thirty minutes, and then set aside for twenty-four hours. The mixture is now shaken out three times with petroleum spirit to remove fat, the extracts, after separation, being filtered in a Gooch's crucible, and the precipitate itself then rinsed on to the filter, washed first with water, then with petroleum spirit, dried at a temperature of 100° to 105° C., and weighed. The weight of the precipitate multiplied by the factor 0·4829 gives the amount of vanillin present.

The method is also applicable to the determination of vanillin in alcoholic extracts, but the alcohol must first be removed by evaporation at a low temperature. W. P. S.

The Determination of Iodine in Iodated Thymols. H. Cormimboeuf. (*Ann. de Chim. anal.*, 1905, vol. 10, pp. 453-454.)—Iodated thymols (aristols) contain from 15 to 45 per cent. of iodine, according to the mode of manufacture. Most of the commercial products are chlor-iodo- rather than di-iodo thymols, and there is a risk of the whole of the halogen present being determined as iodine if the ordinary methods of Carius, etc., be used. The following method is recommended as giving only the iodine and requiring but little material: 0·5 gram of the sample is thoroughly mixed in a mortar with 3 grams of dry sodium carbonate, and the mixture heated gradually in a platinum or nickel crucible until nearly complete combustion of the organic matter, after which it is fused. The mass, when cold, is treated with hot water, the solution filtered from any particles of unburnt carbon, the filtrate mixed with half its volume of ammonium hydroxide, and the iodine precipitated with silver nitrate in the usual way. The filtrate is acidified with nitric acid, a precipitate of silver chloride being obtained in the presence of chlorine. C. A. M.

Oil of False Savin. J. C. Umney and C. T. Bennett. (*Pharm. Journ.*, 1905, vol. 75, pp. 827-829.)—The oil of savin as distilled in this country, in Germany, and in the South of France, varies considerably. This is probably due to the fact that in the latter country the oil is distilled from false savin, *Juniperus Phœnicia* (the common Mediterranean species), whilst in the former the distillers use *Juniperus sabina*. The following results were obtained with three specimens of the oil:

	English. From *J. Sabina.*	German. From *J. Sabina.*	French. From *J. Phœnicia.*
Specific gravity	0·909	0·920	0·892
Optical rotation	+ 68°	+ 42°	+ 4°30′
Esters	47·6 per cent.	36·5 per cent.	9·3 per cent.
Total sabinol	52·1 ,,	48·2 ,,	17·1 ,,
Solubility in 90 per cent. alcohol	1 in 1	1 in 1	1 in 5
Portion distilling:			
Below 155° C.	nil.	nil.	18 per cent.
165° C.	nil.	nil.	64 ,,
175° C.	14 per cent.	nil.	68 ,,
180° C.	23 ,,	4 per cent.	80 ,,
200° C.	48 ,,	29 ,,	82 ,,
220° C.	62 ,,	49 ,,	84 ,,
230° C.	80 ,,	60 ,,	86 ,,

The oil from *J. Phœnicia* contains over 75 per cent. of pinene, the hydrochloride of which melts at 125° C., and the nitrosochloride, after one recrystallization, at 107° C. A sesquiterpene, cadenine, is also present in the oil. W. P. S.

Examination and Assay of Aloes. L. v. Itallie. (*Pharm. Weekblad*, xlii., 553; through *Pharm. Journ.*, 1905, vol. 75, p. 554.)—The author considers that Tschirch's method (Analyst, 1905, xxx., 249) is open to objection, and modifies it as follows: Five grams of the powdered aloes are warmed with 5 c.c. of methyl alcohol until a homogeneous liquid is obtained; 30 c.c. of chloroform are then added, the mixture is shaken for five minutes, and allowed to stand until clear. The solution is decanted, evaporated to dryness, and the residue again dissolved in methyl alcohol and precipitated. This treatment is repeated a third time. By the use of this method Cape aloes were found to contain from 18 to 43 per cent. of resin, and Curacao aloes from 11 to 21 per cent. Objection is also raised to Tschirsh's proposed colorimetric determination of aloin by Schouteten's reaction (green fluorescence with borax solution) on the ground that it is far too subjective, the author finding 100 per cent. of aloin in a sample of aloes. Determination of aloin by precipitation, as tri-bromo-aloin, succeeded with pure aloin, but indicated 71 per cent. of aloin in

Cape aloes and 72 per cent. in Curacao, which results the author considers to be much too high. W. P. S.

Analysis of Powdered Extract of Nux Vomica. W. H. Lenton. (*Pharm. Journ.*, 1905, vol. 75, p. 864.)—The following process is particularly applicable to the analysis of the powdered extract of nux vomica, and is similar to Bird's method (*Pharm. Journ.*, xi., 4, 574), dilute alcohol being used as a solvent for the powder, and a mixture of ether and chloroform for the actual extraction. Ten c.c. each of ether and chloroform are placed in a dry separating funnel, 2 grams of the powder are introduced, then 5 c.c. of 90 per cent. alcohol and 3 c.c. of concentrated solution of ammonia are added, and, finally, 5 c.c. of water. The whole is well shaken for about one minute, allowed to settle, and the ether-chloroform layer drawn off into another separating funnel, where it is shaken with 5 c.c. of 10 per cent. ammonium carbonate solution. After shaking the ether-chloroform once more with ammonium carbonate solution, the former is drawn off and placed aside. The alkaline mother-liquor is twice again shaken out with 20 c.c. of the ether-chloroform mixture, and the extract washed with the ammonium carbonate used for the first portion drawn off. The mixed ether-chloroform solutions are then treated as usual for the determination of strychnine. W. P. S.

ORGANIC ANALYSIS.

Studies on Formaldehyde in Aqueous Solution. F. Auerbach. (*Arbeit. Kaiserl. Gesundheitsamte*, 1905, vol. 22, pp. 1-46.)—The formaldehyde solutions employed in the experiments described were prepared by subliming tri-oxymethylene in an atmosphere of nitrogen, and collecting the vapours in water. For the determination of formaldehyde the sulphite method of Lumière and Seyewetz (ANALYST, 1904, xxix., 288) was found to be very useful, but for small quantities the iodine method was preferred. The results of determinations of the molecular weights of solutions containing varying amounts of formaldehyde lead the author to conclude that formaldehyde exists in aqueous solutions for the greater part in a hydrated or polymerized form. As the temperature is increased the simple molecule predominates. On distilling formaldehyde solutions of any strength whatsoever the distillate is always poorer and the residue richer in formaldehyde than the original solution. This fact should be taken into consideration in the detection and determination of formaldehyde. It is further shown that the boiling-point at normal pressure of formaldehyde solutions falls from 100° to 99° C. as the concentration increases. W. P. S.

Distinction between Phenol and Cresols. C. Arnold and G. Werner. (*Apoth. Zeit.*, xx., 925; through *Pharm. Journ.*, 1905, vol. 75, p. 837.)—The following reactions are suitable for the identification of phenol and cresols :

Phenol.—To 10 c.c. of the solution to be tested are added 10 c.c. each of potassium hydroxide solution and alcohol and 1 drop of aniline. After shaking the mixture, 5 drops of hydrogen peroxide and 10 drops of sodium hypochlorite are added and the whole again shaken. Phenol gives a transient dirty red coloration, changing to yellow; *o*-, *m*-, and tricresol, a violet, changing at once to green; *p*. cresol, a violet, which at once disappears.

o-Cresol.—With ferric chloride *o*. cresol gives a blue colour, rapidly changing to green; phenol, *m*. cresol, and tricresol, a violet; *p*. cresol, a blue colour.

m-Cresol.—On warming with a little phthalic acid and 5 drops of sulphuric acid *m*-cresol gives a cherry-red, phenol and tricresol a dark red, *o*. cresol a cherry-red, *p*-cresol an orange colour. When diluted with water and rendered alkaline with sodium hydroxide phenol gives a magenta, *o*-cresol and tricresol a violet-red, *m*-cresol a bluish violet, and cresol a yellowish colour.

Cresol.—A dilute ammoniacal solution of cresol when boiled and treated with bromine water gives no coloration. Phenol and *o*-cresol treated in this way give a blue colour; *m*-cresol and tricresol a bluish-green.

If a trace of potassium nitrate be added to a solution of a little of the substance in sulphuric acid, a dark-red colour is obtained in the case of cresol, all the others giving an emerald-green coloration. When diluted with water and treated with an excess of ammonia, cresol gives a yellow colour, all the others a green. W. P. S.

On the Inflammability of Celluloid Articles (according to experiments made by Fr. Gervais, director of the Laboratory of the Ministry of Finance, St. Petersburg.) **J. Bronn.** (*Zeits. angew. Chem.*, 1905, xviii., 1976.)—It is shown that when heated to 100° C. (for instance, by contact with a steam-heater) articles made of celluloid decompose exothermically. Although the quantity of heat developed is not sufficient to set the celluloid itself on fire, it will cause the paper used for packing to smoulder, a slight draught of air being then sufficient to cause the whole mass to ignite. The temperature of ignition of celluloid appears to be fairly high, ranging from 355° to 457° C. in the case of four articles examined, that of pyroxylin being only 130° C. White celluloid (imitation ivory) is more difficult to ignite than the other kinds. Celluloid articles may now only be sent to Russia when packed in wooden or metal cases. A. G. L.

The Detection of "Blown" Fatty Oils in Mixtures with Mineral Oil. J. Marcusson. (*Chem. Rev. Fett- u. Harz. Ind.*, 1905, xii., 290-293.)—"Blown" rape and cotton-seed oils are stated to be the two preparations of this kind most used in lubricating mixtures. Their presence cannot be detected by determination of the iodine value or molecular equivalent of the fatty acids. "Blown" cotton-seed oil does not give Halphen's or Milliau's reactions, though it gives the brown coloration with nitric acid. The latter reaction, however, is also given by "blown" rape oil. The author relies upon the following tests to distinguish between the two "blown" oils: (1) The odour of the mixture and of the fatty acids, recalling that of the unblown oils. (2) The consistency of the fatty acids—oily in the case of "blown" rape oil, semi-solid in that of "blown" cotton-seed oil. (3) The behaviour of the respective lead salts towards ether. Thus the amounts of fatty acids from the lead salts insoluble in ether ranged from 1·2 to 20·6 per cent. in the case of five "blown" rape oils, and 32·9 to 45·8 in the case of two cotton-seed oils. The difference was more marked when the solubility of these fatty acids in petroleum spirit was compared, the rape oils yielding 0 to 8·7 per cent. and the cotton-seed oils 23·3 to 32·5 per cent. The fatty acids from the insoluble salts of the "blown" cotton-seed oil melted at 54° to 56° C., whilst those of the "blown" rape oil were oily or semi-solid. The following table gives results thus obtained, the amounts being calculated on the quantity of oil used in the preparation of the lead salts:

Kind of Oil.	Fatty Acids separated from Lead Salts insoluble in Ether.		
	Total Fatty Acids.	Fatty Acids soluble in Petroleum Spirit.	Fatty Acids insoluble in Petroleum Spirit.
	Per Cent.	Per Cent.	Per Cent.
Commercial "blown" rape oil ...	1·2	1·2	0·0
Ditto	14·5	5·7	8·8
Rape oil "blown" in laboratory...	20·6	8·7	11·9
Commercial "blown" cotton-seed oil	32·9	23·3	9·6
Cotton-seed oil "blown" in laboratory	45·8	32 5	13·3

C. A. M.

On the Determination of Sulphur in Liquid Fuels (Petroleum, Oil, etc.). J. Matwin. (*Zeits. angew. Chem.*, 1905, xviii., 1766.)—The author recommends a modification of Drehschmidt's method in place of that of Goetzl (Analyst, 1905, xxx., 376) for the determination of sulphur in liquid fuels, as much larger quantities (50 to 100 grams) may be used: The fuel is burnt in a small lamp provided with a wick, the products of combustion being led through a 5 per cent. solution of potassium hydroxide contained in three wash-bottles. The difference in weight of the lamp before and after the experiment gives the quantity of fuel burnt. No test analyses are given.

A. G. L.

Determination of Sulphur in Petroleum and Bituminous Minerals. F. C. Garrett and E. L. Lomax. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 1212.)—A convenient quantity (0·7 to 1·5 gram) of the substance is intimately mixed in a small crucible with 3 or 4 grams of a mixture of 4 parts of pure lime with 1 of anhydrous sodium carbonate, and the crucible completely filled with this mixture. A larger platinum crucible is placed over the small one (mouth downward), the whole inverted, and the space between the two crucibles filled with the lime-soda mixture. The apparatus is then placed in a muffle furnace heated to bright redness, the mouth of the crucible being covered with a thick pad of asbestos board, which prevents distillation from the inner crucible before the mixture in the outer crucible is heated, due to radiation from the roof of the muffle, and the pad may be removed as soon as a flame appears. The roasting should be continued for two hours, after which the mixture is brought into water, any sulphide oxidized by bromine, and the solution acidified, filtered, and precipitated by barium chloride in the usual way, the solution being allowed to stand on the water-bath for twenty-four hours before filtration, if the amount of sulphur is small. (*Cf.* ANALYST, 1905, xxx., 418.)

W. H. S.

Estimation of Tannin as Strychnine Tannate. S. R. Trotman and J. E. Hackford. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 1096.)—The present absorption methods of determination are very unsatisfactory, even when the hide powder is replaced by collin, and the authors have investigated a large number of bodies with the object of obtaining a compound of tannin which could be readily precipitated and weighed. Metallic salts proved useless, precipitation being incomplete, and after experimenting with many organic bases, including phenylhydrazine and various primary and secondary amines, the alkaloids were tried, and strychnine, one molecule of which combines with one molecule of tannin, finally adopted, as it does not precipitate gallic acid, and its tannate is highly insoluble in water. Sufficient material is extracted with alcohol in a Soxhlet extractor to yield about 0·5 gram tannin, the solution evaporated to 50 c.c, transferred to a 100 c.c. flask, and made up to the mark with water, thus precipitating any resins and similar bodies. These are filtered off through a dry Gooch crucible, and the tannin estimated in the filtrate, 25 c.c. of which are placed in a 250 c.c. flask, diluted with water, the cooled strychnine solution (prepared by dissolving 0·25 gram strychnine in 50 c.c. alcohol, and mixing with an equal volume of water) added, and the solution made up to 250 c.c. This method of treatment is essential, as otherwise the precipitate is difficult to filter. The contents of the flask are now filtered through a weighed Gooch crucible of platinum or porcelain, having a diameter at the bottom of 1 to 2 inches, with a thin mat of asbestos over the bottom. After filtration the tannate is partly air-dried, and the dehydration completed in a vacuum oven heated to about 60°. The soluble non-tannins are determined by evaporating 25 or 50 c.c. of the filtrate from the resinous matter. The amount of tannin found by the method is invariably less than that obtained by the use of hide powder or collin. W. H. S.

The Detection of Biliary Pigments in Urine. L. Grimbert. (*Journ. Pharm. Chim.*, 1905, xxii., 487-492.)—The following method combines the principles of the methods of Hammarsten and Salkowski, but it is claimed that it is more simple than either: Ten c.c. of the urine are shaken with 5 c.c. of a 10 per cent. solution of barium chloride, and the mixture subjected to centrifugal force. The precipitate, consisting of the sulphate, phosphate, and bilirubinate of barium, is mixed with 4 c.c. of 90 per cent. alcohol containing 5 per cent. of hydrochloric acid, and heated for about a minute on the water-bath, after which the tube is allowed to stand. If the supernatant liquid over the deposit is bluish green or dark green the presence of biliary pigments is indicated, whilst in their absence the liquid is colourless. If there is a slight brownish tint the hydrochloric acid may have been insufficient to oxidize the barium bilirubinate, and in that case, and that case only, 2 drops of hydrogen peroxide (10 vol. solution) are added and the tube again heated on the water-bath, after which the green colour will appear. If the brown tint continues after the addition of the hydrogen peroxide the presence of certain altered products of biliary pigments is indicated. These are only met with in urines that have been allowed to stand for some time. In certain pathological urines the separation of the barium bilirubinate is promoted by the addition of a few drops of a 10 per cent. solution of sodium sulphate. C. A. M.

Estimation of Naphthalene in Coal Gas. C. J. D. Gair. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 1279.)—Three methods are described, the first a slight modification of Colman and Smith's, in which a measured volume of gas is passed through three Woulff's bottles, each containing exactly 500 c.c. of $\frac{N}{20}$ picric acid, the contents of the bottles mixed together in a 20-ounce flask, and heated on the water-bath at 60° C. until the precipitated naphthalene and naphthalene picrate have entirely dissolved, the flask being closed with a cork through which a glass tube dips into a beaker containing a small quantity of picric acid from the Woulff's bottles. This is afterwards returned to the flask and its contents cooled, when naphthalene picrate crystallizes out, is dried *in vacuo* or in a warm room, and weighed, whence the naphthalene may be calculated; or an aliquot part of the clear residual picric acid may be titrated with $\frac{N}{10}$ sodium hydroxide, using lacmoid as indicator, showing the amount of picric acid taken up by the napthalene. This method, though devised for pure coal gas, is applicable to impure gas if the ammonia be first removed by passing the gas through oxalic acid before entering the picric acid solution.

The second method, due to Somerville, consists in passing a known quantity of the gas through three glass tubes, 7 inches long and 1 inch wide, fitted up as Woulff's bottles, and each containing 35 c.c. of 70 per cent. alcohol. The alcoholic solutions are then mixed in a flask, oxalic acid added if necessary, until the ammonia is neutralized, as shown by litmus-paper, and filtered, the filter-paper being thoroughly washed with dilute alcohol. About 500 c.c. of concentrated picric acid are then added, the liquid agitated, and allowed to stand for half an hour, when all the naphthalene picrate will have separated, and may be filtered, slowly dried, and weighed.

A third method, devised by the author, depends on the solubility of naphthalene in acetic acid. A known volume of gas, usually not exceeding 3 to 6 cubic feet, is passed through 350 c.c. of acetic acid (specific gravity 1·044), contained in two Woulff's bottles, a small Woulff's bottle with 150 c.c. of picric acid being placed after the acetic acid to catch any unabsorbed naphthalene. The contents of all three bottles are mixed in a flask, and 500 c.c. of concentrated picric acid solution added, when the naphthalene picrate separates out at once in large flocculent masses, which are easily filtered off, dried, and weighed. W. H. S.

INORGANIC ANALYSIS.

The Determination of Bismuth, and its Separation from Copper, Cadmium, Mercury, and Silver. A. Staehler and W. Scharfenberg. (*Berichte*, 1905, xxxviii., 3862-3869.)—The method described is based upon the quantitative precipitation of bismuth by sodium phosphate, even in the presence of hydrochloric acid. From 10 to 20 c.c. of the solution of the bismuth salt (0·1 to 0·2 gram of bismuth) are diluted to 300 or 400 c.c., and heated to boiling, any precipitate of basic salt being redissolved by the addition of the smallest possible quantity of nitric acid. The boiling liquid is treated with a boiling 10 per cent. solution of trisodium phosphate, the quantity required depending upon the amount of free acid present. In the presence of much hydrochloric acid a larger addition of sodium phosphate is necessary. If the solution has become alkaline, it must again be rendered acid by means of a little nitric acid. After boiling for some time the precipitate is allowed to subside, the supernatant liquid tested with sodium phosphate, and if the precipitation be complete, the precipitate is collected while hot in a Gooch's crucible, washed with hot 1 per cent. nitric acid containing a trace of ammonium nitrate, dried at 120° C., ignited for five or ten minutes over a Bunsen-burner, and weighed as $BiPO_4$. The tabular results quoted agree within fractions of a mgm. with the theoretical amounts.

Separation of Bismuth and Copper.—The bismuth is precipitated as above described, whilst the copper is precipitated from the filtrate by means of hydrogen sulphide, and weighed as cuprous sulphide (Cu_2S). Electrolytic determination is unsatisfactory in the presence of hydrochloric acid.

Separation of Bismuth and Cadmium.—After separation of the bismuth as phosphate, the cadmium can be determined by electrolysis of the ammoniacal filtrate after the addition of potassium cyanide, provided hydrochloric acid be absent. In the presence of hydrochloric acid it is best to precipitate the cadmium as sulphide, which is then dissolved in dilute nitric acid, and the solution electrolyzed after the addition of potassium hydroxide and potassium cyanide.

Separation of Bismuth and Mercury.—The filtrate from the bismuth phosphate is treated with a large amount of hydrochloric acid, and then with ammonia in excess, any precipitate formed being redissolved in hydrochloric acid, and the solution again rendered alkaline with ammonia. The liquid is next heated to the boiling-point, and immediately treated with hydrogen sulphide. After five to ten minutes the mercury sulphide separates out uncontaminated with free sulphur. It is collected on a Gooch's filter, washed with hot water, alcohol, and ether, dried at 105° C., and weighed.

Separation of Bismuth and Silver.—The bismuth can obviously only be separated in the absence of hydrochloric acid. The silver in the filtrate from the phosphate can be determined by the usual methods.

Separation of Lead and Bismuth.—It is suggested that a method of separation may be based on the precipitation of the two metals as phosphates, and the removal of the lead by boiling the precipitate with nitric acid (1 per cent.). Lead is quantitatively precipitated from a neutral solution by means of sodium phosphate; but the precipitate consists of at least three different phosphates, which are converted, however, by long-continued boiling into the tertiary phosphate.

C. A. M.

On the Precipitation of Metallic Gold. P. E. Jameson. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1444.)—By the following method metallic gold may be precipitated in one or two minutes in a form resembling silver chloride. A stick of potassium nitrite weighing about 5 grams is placed in a solution of 1 gram of gold chloride in 30 c.c. of water, and 5 c.c. of concentrated sulphuric acid are at once added. As soon as the brisk reaction which takes place is over, another piece of potassium nitrite of the same size as the first is added, and the solution stirred until the reaction is over. The gold will then be found at the bottom of the clear liquid in the form of dark brown nodules, which, after drying, are more yellow than ignited cadmium oxide. A. G. L.

Estimation of Platinum. Nordenskjold. (*Chem. Zeit. Rep.*, 1905, xxix., 293.) --Platinum is usually estimated as the metal by precipitating with sodium formate or with zinc. The former method can be carried out by treating the platinum solution with 2 grams sodium formate and a little ammonium acetate, diluting to 200 c.c., warming until the evolution of carbon dioxide slackens, and then heating for twelve hours. The platinum is apt to pass through the filter-paper, but this can be prevented by adding a little nitric acid. The second method gives a granular precipitate of platinum. Magnesium is preferable to zinc. About 17 cm. of magnesium ribbon should be used per gram of platinum, and dilute solution used. Excess of magnesium is removed by hydrochloric acid; any magnesium oxychloride is at the same time dissolved out. The two methods give concordant results. For iridium, ruthenium, and rhodium a slight modification is necessary, as these metals are sensibly attacked by acids in the precipitated form; only sufficient excess of magnesium to form oxychloride is used, the residue is heated, whereby the metals mentioned are rendered insoluble in acids, and the oxychloride of magnesium dissolved out with acid.

To estimate platinum in a mixture of this group, the heated metal powder is digested with aqua regia, when platinum (and palladium) go into solution.

E. K. H.

Note on an Antimonial Pigment for Enamels. P. Rasenack. (*Arbeit. Kaiserl. Gesundheitsamte*, 1905, vol. 22, pp. 653, 654.)—The author finds that a substance sold under the name of "Leukonin" consists almost entirely of sodium metantimoniate, and that it is soluble to a considerable extent in water and in solutions of sodium chloride, sodium carbonate, tartaric acid, citric acid, etc., antimony in every case going into solution. The author suggests that enamels made with this substance may yield antimony to weak acid liquids. W. P. S.

The Use of Potassium Periodate in the Detection of Manganese, Cobalt, and Zinc. S. R. Benedict. (*Amer. Chem. Journ.*, 1905, vol. 34, pp. 581-585.) —On adding a solution of potassium periodate to a solution of a manganous salt a deep-red precipitate (or coloration with very dilute solutions) is immediately produced. This precipitate is soluble in hydrochloric acid, and is converted by sodium peroxide into a black hydrated manganese dioxide. As a reagent, a $\frac{N}{10}$ solution of the potassium salt is capable of detecting 1 part of manganous chloride in 10,000, the test being more sensitive in a warm ammoniacal solution than in a neutral warm solution. Zinc does not interfere with the reaction. Solutions of pure zinc salts yield a white periodate, but the presence of ammonium chloride and ammonium hydroxide prevent the formation of this, unless there is a very large amount of zinc. On boiling the filtrate from the precipitated manganese the zinc is precipitated. Conversely, a solution of manganese chloride may be used for the detection of periodate in the presence of iodate, iodide, etc. Iodates give no coloration with manganese in the presence of ammonium chloride and hydroxide.

Solutions of pure nickel salts react with potassium periodate to give a light-green precipitate, which becomes slightly grey on boiling, whilst cobalt salts yield a dark-brown precipitate, which becomes greenish on adding an excess of the reagent, and dissolves, forming a greenish-black solution on heating. If cobalt and nickel are present together, the precipitate will become more or less olive green on boiling, and the test is capable of detecting as little as 0·1 per cent. of the former. It is advisable, however, to make a comparative test with a solution of a pure nickel salt of the same strength. If the precipitate turns black, either in the cold or on boiling, traces of manganese are present. C. A. M.

Separation of Iron and Zinc by Means of Ammonia. W. Funk. (*Zeits. angew. Chem.*, 1905, xviii., 1687.)—In the separation of iron and zinc by means of ammonia, the author shows that good results can be obtained if a large excess of ammonia is used in the presence of much ammonium chloride, the precipitate being washed with a 5 per cent. solution of ammonium chloride, and the precipitation being carried out twice. Practically the whole of the zinc is then found in the two filtrates. A. G. L.

Estimation of Metallic Iron in Presence of Ferrous and Ferric Oxides. B. Neumann. (*Chem. Zeit. Rep.*, 1905, xxix., 310.)—Metallic iron can only be directly estimated in presence of ferrous oxide when no ferric oxide is present, otherwise the latter is reduced to ferrous oxide, using up hydrogen. The author therefore proposes the following indirect method: the substance is very finely powdered, and if only the content of metal is required, this is simply extracted with a magnet. In other cases the finely-divided substance is warmed with a measured quantity of a standard copper sulphate solution, and the precipitated copper directly estimated or, better, calculated from a determination of the copper left in the solution. This gives the metallic iron. Next the hydrogen given off on treatment with dilute sulphuric acid is measured. This represents only part of the hydrogen, some being used in reducing ferric oxide to ferrous oxide. The solution is then titrated with standard permanganate, which gives the original ferrous oxide dissolved and the reduced oxide. From these figures the original content of ferrous oxide can be calculated. The total iron is then estimated in the usual manner, and the ferric oxide obtained by difference. E. K. H.

Determination of Zinc in Zinc Aluminium Alloys. R. Seligman and F. J. Willott. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 1278.)—A rapid method for the approximate determination of zinc (within 0·2 per cent. of the gravimetric result) consists in dissolving the alloy in caustic soda, precipitating the zinc as sulphide, redissolving in hydrochloric acid, and titrating the zinc with potassium ferrocyanide. Half a gram of the drilled alloy is dissolved in a 400 c.c. beaker in 25 c.c. of a 25 per cent. caustic soda solution, the liquid warmed till rapid evolution of gas takes place, and allowed to stand until solution is complete—usually five to ten minutes. The solution is then diluted to 300 c.c. with boiling water, any undissolved iron, copper, lead, tin, or nickel allowed to settle, and the clear liquid poured off, the residue being washed twice by decantation. This residue generally contains a small amount of zinc, which is removed by dissolving in a few drops of concentrated hydrochloric acid, diluting to 20 c.c., neutralizing with caustic soda, and adding 2 c.c. of the latter solution in excess. After warming the precipitated hydroxides are filtered off and washed, and the filtrate added to the main bulk of the zinc and aluminium solution. The zinc is now precipitated by sulphuretted hydrogen, which is passed till a skin forms at the point where the bubbles of gas burst, the precipitate allowed to settle, the clear liquid decanted, and the precipitate thrown upon a filter, whence it is dissolved in 8 c.c. hydrochloric acid, washed into the beaker used for precipitation, and diluted to 250 c.c. with boiling water. The presence of a small amount of alumina in the zinc is harmless, so that it is unnecessary to wash the precipitated sulphide. Five grams of ammonium chloride are then added, and the zinc titrated with a solution containing 22 grams of potassium ferrocyanide per litre, using uranium nitrate or acetate or ammonium molybdate as indicator. For the spot-tests a slab of paraffin wax or a porcelain plate covered with a thin coat of wax is recommended. W. H. S.

The Determination of Barium as Chromate, and its Separation from Strontium and Calcium. A. Skrabal and L. Neustadtl. (*Zeit. anal. Chem.*, 1905, vol. 44, pp. 742-755.)—The authors from a study of the different methods find that good results are obtained by precipitating the barium by means of ammonium bichromate from a neutral or slightly acid solution in the presence of ammonium acetate. The precipitation may be made from a hot or cold solution, but the filtration and washing of the precipitate must be done in the cold. Barium can be separated from calcium with approximately correct results by means of a single precipitation with ammonium bichromate from a cold, very dilute acetic acid solution in the presence of ammonium acetate. Better results are obtained, however, by a double precipitation. It is impossible to effect a complete separation of barium from strontium by means of a single precipitation with ammonium bichromate. The following combined method is recommended for the separation of barium from calcium and strontium: The neutral or slightly acid solution of the salts (about 0·135 gram of the respective oxides) is mixed with ammonium acetate in excess (10 c.c. of a solution containing 300 grams per litre), brought to the boiling-point, and treated with ammonium bichromate solution (5 c.c. of a solution containing 100 grams per litre), added drop by drop, with continual shaking. The precipitate is allowed to subside, and when cold washed by decantation with a dilute solution of ammonium acetate (20 c.c. of the above solution diluted to a litre) until the washings passing through the filter are colourless. The small amount of the precipitate that has been retained by the filter is dissolved in warm dilute nitric acid, the solution and washings returned to the original beaker, and sufficient nitric acid added to dissolve the whole of the precipitate. The clear solution is now treated drop by drop with ammonium hydroxide until the first signs of a permanent turbidity appear, after which ammonium acetate is added (10 c.c. of the strong solution), and the liquid boiled and allowed to cool gradually. The precipitate is washed by decantation and on the filter with cold dilute ammonium acetate solution, then dried, ignited in a platinum crucible, and weighed. Strontium and calcium are then determined in the united filtrates in the usual manner. C. A. M.

The Determination of Titanium in Soil and the Ash of Plants. H. Pellet and C. Fribourg. (*Ann. de Chim. anal.*, 1905, vol.10, p. 413-416.)—I. *Direct Colorimetric Method.*—Half a gram of the dry earth, or 2·5 grams of ash, are introduced into a platinum crucible containing 15 grams of pure hydrofluoric acid and 1 c.c. of sulphuric acid, and evaporated to dryness. The residue is powdered, mixed with 5 grams of potassium bisulphate and fused, and the resulting product taken up with water containing 15 c.c. of sulphuric acid per 100 c.c., at a temperature not exceeding 60° C. The liquid is made up to 100 c.c. (there should be no insoluble residue), 1 to 10 c.c. taken and made up to 100 c.c. with distilled water, treated with 5 c.c. of hydrogen peroxide (12 volume solution), and compared colorimetrically with solutions of pure titanic acid containing 0·1 to 1·0 gram per litre.

II. *Gravimetric Methods.*—In the case of soils containing about 2 per cent. of titanic acid, 3 grams of the dried and finely-powdered sample are introduced little by little into a platinum crucible containing 30 grams of hydrofluoric acid, the mixture evaporated to dryness on the water-bath, after the addition of 3 c.c. of sulphuric acid, and the residue powdered and fused with 15 grams of potassium bisulphate. After cooling, the mass is powdered and dissolved in 200 to 250 c.c. of water at a temperature of about 60° C., and the solution, when cold, made up to 300 c.c. and filtered; 250 c.c. of the liquid is next transferred to a 400 c.c. beaker, and 40 c.c. of the remainder titrated with a solution of potassium hydroxide, 10 c.c. of which neutralize 5 grams of potassium bisulphate. From the result can be calculated the amount of potassium hydroxide solution to be added to the 250 c.c., so as to leave 5 grams of potassium bisulphate still unneutralized. After this addition, the liquid is treated with 50 c.c. of a freshly-prepared solution of sulphurous acid (1·020 to 1·025 specific gravity) and boiled for two hours, in the course of which two fresh additions of 50 c.c. each of the sulphurous acid solution are made. It is then filtered, and the precipitate washed with boiling water and ignited, the residue being nearly pure titanic acid, with a little phosphoric acid. It is fused with pure potassium carbonate, the mass taken up with boiling water, and the insoluble portion washed with a 2 per cent. solution o potassium carbonate. The titanic acid remains behind in the form of an insoluble titanate, and the traces that pass into solution can be determined by a blank experiment with pure titanic acid. This titanate is ignited, fused with 1 gram of potassium bisulphate, and treated as above described, the residue of titanic acid being ignited and weighed.

In the case of soils containing less than 1 per cent. of titanic acid, 2 portions of 5 grams each of the finely-powdered and dry sample are ignited in separate platinum crucibles to destroy organic matter, and then fused with a mixture of 10 grams of sodium carbonate and 10 grams of potassium carbonate. The products of the fusion are taken up with very dilute hydrochloric acid, so as to form one solution, the silica separated by evaporating the liquid to dryness and igniting the residue, this being again taken up with dilute hydrochloric acid and the liquid filtered. The residual silica is treated with a mixture of hydrofluoric acid and sulphuric acid, which leaves a sulphated residue, whilst the filtrate is treated with ammonia, and the resulting precipitate separated from the filter and dried (the filter-paper being ignited alone). The united residue from the silica and ammonium precipitate is then fused with 15 to 20 grams of potassium bisulphate, and the titanic acid determined as described.

In the case of the ash of plants containing about 0·2 per cent. of titanic acid, 50 grams are treated with dilute hydrochloric acid, the silica separated, and the titanium determined in the filtrate. C. A. M.

Rapid Method for the Valuation of Fluor-spar. A. W. Gregory. (*Chem. News*, 1905, vol. 92., pp. 184-185).—The following method may be found useful for the determination of calcium fluoride in fluor-spar when carbonates and silica are present in the mineral, all the determinations being carried out on the sample previously dried at 120° C. The carbon dioxide is determined by heating 2 grams of the sample to a red heat, until no further loss in weight takes place. Two grams of the sample are treated in a platinum basin with pure hydrofluoric acid, evaporated, ignited, and weighed. The calcium carbonate is thus converted into fluoride, and the loss in weight obtained is due to the silica present after allowance has been made for the quantity of carbonate previously found. (When the silica is present partly as silicate, this determination is not strictly accurate.) Another quantity of 2 grams of the sample are now heated in a platinum basin with concentrated sulphuric acid, hydrofluoric acid being first added if the amount of silica present be large. The excess of sulphuric acid is then driven off and the residue ignited and weighed. In this operation there will be an increase in weight, due to the conversion of calcium carbonate and fluoride into sulphate, and a loss of any silica present. The amounts of carbonate and silica being known, the fluoride may be calculated.

W. P. S.

Electrolytic Calcium. Joseph H. Goodwin. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1403.)—Metallic calcium can be easily and quickly prepared from calcium chloride by using the apparatus described. It consists essentially of a hollow cylinder of Acheson graphite, which is used as anode, the bottom being formed of a cooling copper coil, insulated from the graphite by asbestos. The cathode is a ⅜-inch iron rod mounted on a screw arrangement, by which it can be raised or lowered. The calcium separated at first acts as cathode during the remainder of the operation, and, by gradually raising the iron cathode, sticks of metallic calcium are easily obtained. The reaction is started by filling the anode with cold calcium chloride, and producing an arc between the cathode and anode; as the calcium chloride melts, the cathode is lowered into it until electrolysis takes place, the heat generated being sufficient to keep the salt fused. The temperature must be kept within rather narrow limits, otherwise the calcium separates in a spongy instead of a coherent form. As, with the heavy currents used, the asbestos does not completely insulate the copper coil from the anode, a cell is inserted between the copper and the graphite so as to cause a small current (0·04 ampère) to flow in the other direction; this device effectually prevents contamination of the calcium by copper. In six experiments, with voltage varying from 14 to 22, and ampèrage from 105 to 185, a current efficiency of 21·5 to 41·9 per cent. was obtained, the average figures being: Volts, 17·7; ampères, 163·0; efficiency, 26·6 per cent. In six hours about 150 grams of calcium can be obtained. In one experiment the separated metal had the following composition: Ca, 98·00; Si, 0·03; Fe, 0·02; Al, 0·03; Mg, 0·11; Cl, 0·90; and O, by difference, 0·91 per cent. This sample had a conductivity of 3·43 microhms per c.c. at 0° C., the temperature coefficient being 0·00457, and a tensile strength of 8,710 lbs. per square inch, the elongation being 23·0 per cent. for 1 cm., 15 per cent. for 2 cm., 11·0 per cent. for 3 cm., and 6·6 per cent. for 5 cm. Calcium was found to be harder than sodium, lead, or tin, almost as hard as aluminium, but softer than zinc, cadmium, or magnesium. Its specific gravity was found to be 1·5446 at 29·2° C.

To clean the metal, after breaking off most of the calcium chloride, it is placed in 95 per cent. alcohol for some time, the loss owing to reaction with water and alcohol not being very great. The solid metal can be heated to a red-heat in air without igniting. It is not hardened by quenching in water. At 300° to 400° C. it is as soft as lead. When cold, the bright surface of the metal rapidly dulls in air, but when hot its surface may be polished, and will keep bright as long as the metal remains hot. By placing the hot metal in a bottle heated to 150° C., and closing the bottle while at this temperature, the surface may be preserved quite bright. A number of the cylinders obtained were fused together in a closed iron tube and quickly cooled, when a mass of large reddish-violet cubical cystals was obtained, having the following composition: Ca, 91·28; gangue, 0·03; SiO_2, 0·77; Fe_2O_3, 0·46; Al_2O_3, 0·77; Mg, 0·11; Cl, 1·28; C, trace; N, trace; O, by difference, 5·30. The specific gravity of the crystals was 1·5425 at 28·1° C; they were quite soft, and could be hammered into sheets as thin as paper, often exploding with a slight flame under the impact of the hammer. A. G. L.

Estimation of Percentage of Nitric Acid in the Concentrated Acid by the Specific Gravity. Veley and Manley. (*Chem. Zeit. Rep.*, 1905, xxix., 1207.)—The authors publish some figures and details of their methods of purification, etc., in consequence of the divergence shown between the results published by Lunge and by Winteler.

The authors come to the conclusion that Winteler's acid was not sufficiently pure. They purified their acid as follows: The purest commercial acid was distilled, and the distillate was redistilled over silver and barium nitrates to remove sulphuric and hydrochloric acid. A current of ozonized oxygen was then passed through, and finally the acid was fractionated *in vacuo* at the lowest possible temperature. In this way an acid was obtained containing 99·8 per cent. HNO_3 with only 1 part per million of nitrous acid, 4 per million of sulphuric acid, and 3 per million of halogen acids. This acid was further freed from water by a special apparatus (*Phil. Trans.*, 1890, A. 365), and an acid finally obtained which contained 99·97 per cent. HNO_3.

The authors used for titrating the various dilutions of this acid a sodium hydroxide solution prepared from sodium and steam, and a *N*-sulphuric acid solution standardized against sodium carbonate.

The following table shows the figures obtained by various observers :

Specific gravity :	1·485	1·490	1·495	1·500	1·505	1·510	1·515	1·520
Percentage according to—								
1. Lunge and Rey	87·7	89·6	91·6	94·1	96·4	98·1	99·1	99·7
2. Veley and Manley	87·5	89·6	91·1	94·0	96·5	97·8	98 7	99·8
3. Ferguson	87·6	89·7	91·4	93·7	—	—	—	—
4. Mean of first three series .	87·6	89·6	91·4	93·9	96·4	97·9	98·9	99·8
5. Winteler	86·3	88·2	90·2	91·8	93·6	95·6	97·3	99·7
Difference between 4 and 5 .	− 1·3	− 1·4	− 1·2	− 2·1	− 2·8	− 2·3	− 1·6	—

For further comments on these figures, reference should be made to the original; but the authors express the view that the clearing up of the question lies with Winteler rather than with the other observers. E. K. H.

Estimation of Percentage of Nitric Acid in the Concentrated Acid by the Specific Gravity. H. Pützer.

(*Chem. Zeit. Rep.*, 1905, xxix.)—Compare paper by Veley and Manley, (see previous abstract). He adds his contribution to the figures already published. Great care was bestowed by the author on the purification of the acid. One hundred and fifty grams of this on evaporation in a platinum dish left a scarcely visible residue which was not weighable; the acid was quite free from nitrous acid (when diluted with water (5:1) and 1 drop of $\frac{N}{10}$ potassium permanganate added it remained pink for an hour), sulphuric acid, hydrochloric acid, and iodine.

The author having found the ordinary method of obtaining sodium carbonate from so-called chemically pure sodium bicarbonate unsatisfactory, he therefore prepared $Na_2CO_3.H_2O$ by boiling a solution of the bicarbonate and re-precipitating. This was then heated in a platinum dish by a burner whose flame-tip was about 5 cm. below the bottom of the dish. $\frac{N}{5}$ Na_2CO_3 was used, with methyl orange, very dilute, as indicator. The specific gravity was determined by a hydrometer showing the first three decimal places exactly, and the fourth by estimation.

The author's figures are :

Specific Gravity $\frac{15^\circ}{4^\circ}$ (Vac.)	Pc. HNO_3.	Specific Gravity $\frac{15^\circ}{4^\circ}$ (Vac.)	
1·4878	89·19	1·5018	95·51
1·4908	90·40	1·5033	96·13
1·4923	90·98	1·5043	96·59
1·4943	91·79	1·5058	97·21
1·4958	92·52	1·5073	97·76
1·4973	93·22	1·5103	98·62
1·4988	93·93	1·5128	99·20
1·4998	94·53	1·5163	99·75

By graphic interpolation these figures give results differing uniformly by 0·5 from those of Lunge and Rey.

The author considers that these differences are due to the fact that his acid was even purer than that used by Lunge and Rey, whilst Winteler's results may be rejected, as the acid used was, on his own showing, not pure. E. K. H.

On the Determination of Sulphuric and Nitric Acids in "Nitrous Vitriol." G. Lunge and E. Berl. (*Zeits. angew. Chem.*, 1905, xviii., 1681.)—As the result of determinations made, partly on a synthetic, partly on a commercial acid, the authors recommend that nitro-sulphuric acid should be examined for (*a*) total acidity, (*b*) permanganate consumed, (*c*) total nitrogen by means of nitrometer. The nitric acid may then be calculated by subtracting (*b*) from (*c*), and the sulphuric acid by subtracting (*c*) from (*a*). Gravimetric determinations of sulphuric acid as barium sulphate tend to give high results; determinations of the nitric acid by the "nitron" method easily lead to low values. Evaporation of the nitric acid and titration of the residual sulphuric acid generally gives results slightly too low for the sulphuric acid, and consequently a little too high for the nitric acid.

A. G. L.

Modification of W. Winkler's Method for the Determination of Oxygen in Waters. Hermann Noll. (*Zeits. angew. Chem.*, 1905, xviii., 1767.)—The author believes that with ordinary waters Winkler's method gives more exact results if the correction made by Winkler for the iodine consumed by the organic matter present is *not* made. When a correction must be made, as in the case of sewage effluents, he proceeds as follows: A mixture of 2 c.c. of 40 per cent. sodium hydroxide solution, 2 c.c. of 50 per cent. manganous chloride solution, and 20 c.c. of distilled water is shaken in a large flask until it becomes brown in colour; 50 c.c of concentrated hydrochloric acid are then added, and the whole made up to 300 c.c. with distilled water. Of this solution, 20 c.c. are added to 100 c.c. of the water under examination, and also to 100 c.c. of distilled water. After five minutes 10 c.c. of a 5 per cent. potassium iodide solution are added to each, and the iodine liberated is determined with a solution of sodium thiosulphate, 1 c.c. of which corresponds to 0·1 c.c. of oxygen. The difference between the two values found gives the quantity of iodine taken up by the organic matter in the water, and may be applied as a correction to the results found by Winkler's method. Results obtained in this way on several waters agreed better with gasometric determinations of the oxygen in the waters than the values found using Winkler's method of correction. A. G. L.

APPARATUS.

Alternating Motion for Stirrers. (*Chem. Zeit.*, 1905, xxix., 1208.)—Two stirring wheels are used, which rotate in opposite direction, and are so arranged that one of them is worked by the friction due to a projection on the main wheel during half a revolution of the latter, whilst the other is worked by a second projection on the other half of the main wheel. The two paddles, therefore, work each through the time of a half revolution.

The apparatus is protected, and can be obtained from Franz Hagershoff, Leipzig.

E. K. H.

Note on the Incandescent Mantle as a Catalyst, and its Application to Gas Analysis. J. E. Mason and J. Wilson. (*Proc. Chem. Soc.*, 1905, vol. 21, p. 296.)—Although less effective, the ordinary gas mantle may be used as a substitute for platinized asbestos in the preparation of formaldehyde from methyl alcohol vapour and air, and sulphur trioxide from sulphur dioxide and oxygen. Fragments of mantle in a hard glass or quartz tube may be employed in the place of palladium or palladium-asbestos for the determination of hydrogen and carbon monoxide by combustion with excess of air or oxygen. Methane and mixtures of methane and hydrogen may be determined similarly, the contraction being measured after combustion and subsequent treatment with potassium hydroxide solution. The results agree well with those obtained by the ordinary explosion methods. Good results may also be obtained by passing the gases mixed with oxygen over asbestos heated in a small quartz tube. Hydrogen, and less readily methane, may be determined by passing the gas mixed with oxygen through narrow tubes of heated Jena glass alone. W. P. S.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

Past List of the January Examinations.

Of fourteen candidates who entered for the Intermediate Examination, ten passed A. P. Davson, F. W. Foreman, W. Garsed, T. R. Hodgson, B.A. (Cantab.), T. J. Kirkland, A. Lathwood, B.Sc. (London), B. D. W. Luff, J. F. Reid, H. Stanley, B.Sc. (London), and F. Tattersfield. In the Final Examination for the Associateship (A.I.C.), of five examined in the branch of Mineral Chemistry, two passed: E. R. Bullock, Assoc.R.C.Sc. (London), and T. F. Cowie; of four in Metallurgical Chemistry, two passed: H. J. B. Rawlins, B.Sc. (London), and Thomas Stenhouse, B.Sc. (London), Assoc.R.C.Sc. (London), A.R.S.M.; of four in Organic Chemistry, three passed: W. P. Hayworth, F. H. G. Horsman, B.Sc. (London), and D. Spence, Ph.D. (Jena); and of twelve who entered in the branch of the Analysis of Food and Drugs and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following nine passed: J. T. Cart, B.Sc. (London), C. G. Gates, B.Sc. (London), A. G. Holborow, Miss E. S. Hooper, B.Sc. (London), S. J. Lewis, B.Sc. (London), A. J. C. Lickorish, S. G. Liversedge, Miss E. A. Macadam, F. E. Thompson, Assoc.R.C.Sc. (London). The Examiners in Chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. The Examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., F.R.S., F.I.C.

MARCH, 1906. Vol. XXXI., No. 360.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual meeting of the Society was held on Wednesday evening, February 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Dr. L. T. Thorne and Mr. J. H. B. Jenkins were appointed scrutators of the ballot-papers for election of Officers and Council.

The HON. TREASURER (Mr. E. W. Voelcker, A.R.S.M.) presented his annual report, with the accounts of the Society for 1905.

On the motion of Mr. ARTHUR R. LING, seconded by Mr. JOHN WHITE, the report was unanimously adopted, and a vote of thanks accorded to the Hon. Treasurer for his services during the past year.

The HON. TREASURER, having responded, proposed a vote of thanks, which was unanimously passed, to the auditors, Mr. W. P. Skertchly and Mr. Arthur R. Ling.

A vote of thanks to the Hon. Secretaries, Mr. Alfred C. Chapman and Mr. P. A. Ellis Richards, was proposed by Dr. DYER, seconded by Dr. SCHIDROWITZ, and unanimously passed.

Mr. CHAPMAN acknowledged the vote of thanks.

A vote of thanks to the President and Council of the Chemical Society for their kindness in allowing the Society the use of their rooms at Burlington House during the past year was moved by the PRESIDENT, and unanimously agreed to.

The PRESIDENT delivered his annual Address.

Mr. HEHNER proposed that a very hearty vote of thanks be accorded to the President for his Address, and that his permission be asked to publish the Address in the ANALYST.

Mr. KITTO seconded the resolution, which was carried unanimously.

The PRESIDENT briefly responded.

The scrutators having reported to the President the result of their examination of the ballot-papers,

The PRESIDENT announced that the Officers and Council for 1906 had been elected as follows:

President.—E. J. Bevan.

Past-Presidents (limited by the Society's Constitution to ten in number).—M. A. Adams, F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Thomas Fairley;

W. W. Fisher, M.A.; Otto Hehner; Alfred Hill, M.D.; J. Muter, Ph.D.; Sir Thomas Stevenson, M.D., F.R.C.P.; J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Vice-Presidents.—L. Archbutt, W. J. Dibdin, B. Kitto.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—Alfred C. Chapman, P. A. Ellis Richards.

Other Members of Council.—Arthur Angell; Julian L. Baker; R. Bodmer; M. Wynter Blyth, B.A., B.Sc.; Charles Estcourt; D. Lloyd Howard; J. H. B. Jenkins; E. W. T. Jones; S. Rideal, D.Sc.; Alfred Smetham; W. Lincolne Sutton; John White.

The monthly ordinary meeting of the Society was held on Wednesday evening, February 7, in the Chemical Society's Rooms, Burlington House, immediately after the annual meeting. The President, Mr. E. J. Bevan, occupied the chair.

Certificates of proposal for election to membership in favour of Messrs. S. Dickson, J. Evans, F. Hughes, G. Patterson, and H. Thompson, were read for the second time; and certificates were read for the first time in favour of Messrs. George Craig, F.I.C., 95, Bath Street, Glasgow, analytical and consulting chemist; John B. Gall, F.I.C., A.C.G.I., Knoxland, Erith Road, Belvedere, works chemist to Messrs. Callender's Cable and Construction Company, Limited; H. Norman Hanson, Field Head, Brighouse, assistant to Mr. F. W. Richardson; Bernard W. Methley, 21, Clifton Mount, Rotherham, chemist to Messrs. Steel, Perch and Tozer, Sheffield; Frank Darby Ratcliff, 203, Ashted Row, Birmingham, chemist to Messrs. Holbrooks, Limited; and Frederick Robertson, 128, Wellington Street, Glasgow, analytical and consulting chemist.

Dr. H. W. Wiley was elected an honorary member; and Messrs. Geo. Clarke, Junr., C. A. Hill, B.Sc., and H. J. Horton, were elected members of the Society.

The following papers were read: "Note on Dutch Cheese," by Cecil H. Cribb, B.Sc.; "The Assay of Mercury Ores," by G. T. Holloway, A.R.C.Sc.; "The Purification of Zinc and Hydrochloric Acid," by L. T. Thorne, Ph.D., and E. H. Jeffers; and "The Facing of Rice," by Cecil H. Cribb, B.Sc., and P. A. Ellis Richards.

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THE ASSAY OF MERCURY ORES.

By GEORGE T. HOLLOWAY.

(*Read at the Meeting, February* 7, 1906.)

All the mercury of commerce is obtained by the roasting of cinnabar (HgS) in a current of air. The sulphur burns to the dioxide, and the liberated mercury, together with such native mercury as is frequently associated with the cinnabar, distils over and is collected in condensers and flues. Despite the enormous difficulty which is experienced in condensing the mercury and minimizing loss by leakage and flouring, the cheapness of the process and its comparative simplicity render it possible to treat ores of extremely low value, and the bulk of the mercury now produced has been obtained from ores containing an average of between ½ per cent. and 1 per cent. of the metal. It is true that ore as rich as 7 to 8 per cent. is worked at Almaden, in Spain, the oldest and richest mine in the world; but at Idria (Austro-Hungary), Nikitowka (South Russia), and Napa Consolidated Mines (California) respectively standing second, third, and fourth in order of production, the ore averages well under 1 per cent., as it does also in all other mines whose outputs are sufficiently large to count as factors in the world's production, which is stated to have amounted in 1904 to 3,581 tons of 2,240 pounds.

Cinnabar and native mercury usually occur impregnating or disseminated throughout limestones, dolomites, sandstones, etc. Copper and other ores frequently contain mercury, but not in quantity sufficient for extraction on the large scale. Bituminous matter commonly occurs in association with it, and in a few districts, as in Kweichow (China), antimony sulphide is met with, and seriously interferes with the extraction on account of the flouring of the mercury. The cinnabar and native metal, although frequently found in masses and occasionally in crystals, is practically all so mixed with the gangue and is so friable, and, notwithstanding its great density, so easily lost when "dressed," that no dressing plant has been found capable of treating it profitably, and although hand-picking is occasionally practised, almost the whole of the ore is roasted exactly as it leaves the mine.

The old method of assay consisted in heating a mixture of the ore with iron filings or litharge or lime to redness in a glass tube closed at one end and bent down at the other, which latter dipped into water. A length of magnesite or sodium bicarbonate at the closed end of the tube permitted the generation of a stream of carbonic acid gas to drive the remaining mercury vapour out of the tube after the ore had been decomposed. The mercury was collected, dried, and weighed as metal.

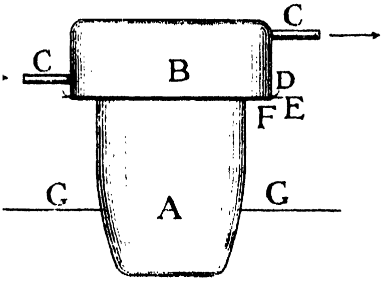
This method is excellent for picked or rich material assaying, say, 20 per cent. or more, and, as stated later, is still probably the most accurate for such material, on account of the large amount of ore which can be taken for assay, but is absolutely useless for the ordinary ore, which probably accounts for 99 per cent. of the world's output. Wet processes are inapplicable to such ore. The well-known Eschka process (*Zeit. für Anal. Chem.*, vol. ii., p. 334), in which a small quantity of the ore was heated with iron filings in a small crucible, and the evaporated mercury was collected on a weighed disc of gold, was therefore introduced. The gold disc was cup-shaped, and was formed with a flange fitting on the crucible top. It contained cold water, which was occasionally renewed during the progress of the test. The mercury became condensed on the gold, which, after being weighed, only required to be heated gently to drive off the mercury ready for the next test.

As regards accuracy, Eschka's process leaves little to be desired, and the use of a strip of gold foil placed at the open end of a small ignition tube of about ¼-inch bore, in which a mixture of iron filings and a mineral or salt containing mercury is heated, forms a most delicate test for mercury.

The presence of mercury to the extent of only $\frac{1}{100}$ of 1 per cent. may thus be proved, and accurate estimations may be made when the total mercury only amounts to $\frac{1}{10}$th per cent.

In 1898, R. E. Chism (*Engineering and Mining Journal*, October 22, 1898, p. 486) introduced a modification in which the gold cup was replaced by a piece of silver foil pressed into shape into the top of the crucible, and his results appear to have been perfectly satisfactory.

The modification about to be described differs from that of Eschka or Chism only in the construction and arrangement of the apparatus, and is shown in the accompanying drawing.



Some difficulty is experienced in fitting the gold cup or silver foil "mercury-tight" to the crucible top, and the cooling water requires frequent renewal, as the collecting power of gold or silver is much reduced when warm. The author therefore employs a flat sheet of "fine" silver, which may be purchased of any thickness at about 3s. 6d. per ounce troy, and which, after annealing by warming to dull redness for a few seconds in a flame, may be rubbed perfectly flat between two surfaces of glass or wood. The silver may conveniently be somewhat thinner than a visiting-card, and is cut into a circular disc, F, about 2 inches in diameter, weighing from 2 to 6 grams according to its thickness. If of the former weight, it can be conveniently weighed on an assay balance, but it must be held up to the light and examined to insure absence of pin-holes, which often occur in thin foil, and which spoil the assay through escape of mercury. The crucible A may conveniently be of the deep form shown, and about 1½ inches wide by 1¾ inches deep, and is ground down perfectly flat at the mouth on emery-paper. An ordinary Berlin porcelain crucible is used. A square plate, E, of silver foil about ½ inch wider than the plate F is used as a cover. The plates E and F are kept cool by a round copper vessel, B, about 2 inches in diameter, with pipes, C, soldered in as shown for entrance and exit of the cooling water. The vessel B consists of a spun cap of copper soldered upon a flat copper disc with a turned up rim, D, which collects such water as occasionally condenses on the cooler. The copper may be about $\frac{1}{16}$ inch thick. The top of a round tin box would serve equally well for the body, but it is advisable to have the bottom of copper on account of its high conductivity. A piece of tin, G, cut out to receive the crucible, localizes the heat around the crucible bottom and rests on an ordinary tripod stand.

The ore to be assayed is heated with iron filings free from grease. The filings are best prepared by heating a quantity of several pounds in a covered crucible to a fair red heat for about an hour. All grease, etc., having thus been driven off or carbonized, the cooled crucible is opened; the top layer of the filings, if at all oxidized, is removed, and the filings are sifted. Those which pass through a 60-mesh sieve and those which pass a 30-mesh sieve are kept in separate stoppered bottles.

The ore is prepared by grinding it very finely, and may be dried before assay if containing no free mercury; but if, as commonly happens, any native mercury is present in a finely-divided state, practically the whole of it may be lost in drying. In that case it must be assayed as received and calculated out on the dry weight. The loss of mercury in drying may be ignored in the determination of *moisture* in any ordinary ore, as it would scarcely affect the result even in the second decimal place.

The amount of ore to be taken for assay depends on its richness, as the silver foil has only a limited power of retaining mercury.

It is advisable to weight out: 2 grams if the ore contains under 1 per cent. of mercury; 1 gram if the ore contains between 1 and 2 per cent. of mercury; 0·5 gram if the ore contains between 2 and 5 per cent. of mercury; 0·25 gram if the ore contains between 5 and 10 per cent. of mercury.

If the ore contains over 10 per cent., correspondingly less should be taken.

The ore is carefully mixed in the crucible with about 10 grams (roughly weighed) of the 60-mesh iron filings, and the mixture is covered with about 5 grams of the 30-mesh filings, even when only so small an amount as 0·25 gram of ore is used. The crucible is placed in the holder G on a tripod stand, the cleaned, flattened, weighed silver disc F is placed on the crucible with the disc E above to keep it clean, the cooler B is placed over all, and a weight is placed on the cooler to prevent displacement. Water supplied through a rubber tube traverses the cooler. A small Bunsen or spirit flame is brought beneath the crucible, the flame being of such a size as to just make the crucible bottom red, but so that the crucible is not red more than $\frac{1}{8}$ inch up the side. The heat is continued for twenty minutes, draughts being carefully excluded, as a draught may easily deflect so small a flame and practically stop the distillation. The iron removes the sulphur and also conducts the heat so that the mercury distils off and is collected by the disc F. After twenty minutes, the flame is removed, and after fifteen minutes' cooling the disc F is removed, dried by placing it in a water-oven for a few minutes, or by holding in tweezers and waving above a flame for a few seconds, cooled, and weighed. The assayer must decide for himself which method to use, and how long to allow for the drying. He should experiment on the disc from one of his assays by damping it, drying, and reweighing until he ascertains the conditions which insure drying without loss of mercury. When the ore contains bituminous matter, as sometimes happens, and as is shown by the smell or by a black or brownish stain on the foil, it is necessary to wash the foil with alcohol and dry as above before weighing.

Until the operator is experienced, it is advisable to reheat the crucible with another weighed piece of foil to insure that all the mercury has been driven off, but this is not usually found to be necessary. If the assay has been satisfactory, the mercury will all be seen as a dull or bright perfectly circular film on the foil, exactly the size of the crucible rim. If it extends beyond, which may occur either from too much ore having been taken, so that the silver cannot properly retain the resultant mercury, or from careless flattening or adjustment of the foil, the assay must be repeated.

When first using the apparatus, the operator should experiment by repeatedly assaying the same sample, varying the temperature and time until he finds the conditions which yield the highest result. Half a dozen such tests, occupying in all not over three or four hours, will suffice for gaining perfect control over the method. By coupling together a number of coolers with rubber tubes and arranging a row of the assays on a bench, one may easily carry on a dozen assays at a time in a confined space. An inverted ½-gallon Winchester, fitted up like an inverted wash-bottle, and having a regulating clip on its indiarubber discharge tube, will suffice for cooling a battery of a dozen assays. The water leaving the last cooler should not feel more than lukewarm.

When the assayer is experienced, he can allow the distillation of one set to continue without watching, while weighing the discs from the previous set.

The special precautions to be observed are: (1) Avoidance of excessive heating of the crucible. The edge of the covering foil E should never feel warm to the fingers. (2) Cleanliness and perfect flattening of the silver foil F. (3) Careful adjustment of the foil and coolers and of the weights on the coolers to insure contact all round the crucible top. (4) Careful drying of the foil. (5) Avoidance of draughts. (6) Allowance of the full time mentioned (fifteen minutes) for cooling before removing the foil, lest uncondensed mercury vapour be lost.

Although gold has a greater affinity for mercury than silver possesses, and is therefore theoretically better, silver has a greater collecting power weight for weight, and a greater conductivity, and has been proved by comparative tests to give at least equally correct results. No comparative figures are given, because the results are identical within the limits of experimental error. Indeed, if anything, the new method gives slightly higher results.

The silver can be used an indefinite number of times, and appears, on account of its greater porosity after prolonged use, to improve.

When elaborate care is used, the method, as tested by diluting down a weighed small quantity of pure cinnabar or pure mercury sulphide or oxide, has been found extremely accurate, but under ordinary conditions a slight loss from leakage may be expected, and the difficulty of sampling such an ore as cinnabar renders the danger of error greater. The cinnabar is extremely heavy and the gangue is usually decidedly light, and the fact that cinnabar commonly occurs as little rich patches in the gangue even in the case of very low-grade ore, and is therefore practically freed during crushing, renders it extremely liable to settle, despite the most careful mixing, and to further accentuate the danger of inaccuracy through sampling. This danger is not great when the ore contains under 10 per cent. of mercury, and may be overcome with care; but when the ore contains 20 per cent. or upwards, it is advisable to distil, say, 10 or 20 grams of the ore in a glass tube with iron filings by the old method already mentioned.

The error due to sampling is, of course, then only one-twentieth or one-fortieth of the probable error on a 0·5 gram sample, and the loss from leakage is practically nil. Such ores, however, seldom require to be assayed, and, as already stated, the old method is practically impossible on average ores. With such low-grade ores it yields the mercury in beads so small that they can scarcely be collected and dried without heavy loss, and it is extremely cumbrous and slow.

On rich ores, the results by the author's method are sometimes too high and sometimes too low, either result being attributable to difficulty in sampling. As, however, the results are usually slightly low on ores containing over 20 per cent. of mercury when done without excessive precautions, the loss is probably due to leakage.

In the case of average ores, the total loss is so small as to be negligible, and duplicate results on a 1 per cent. ore should agree to within 0·05 per cent., a result which cannot be equalled by any wet method.

Experiments have been made in which the iron filings are replaced by reduced iron, or by copper, or by litharge, lime, etc.; but, apart from the extreme convenience of iron filings, the other materials have shown no advantage whatever, and litharge and lime are distinctly less effective.

The author is informed that the method works well with salts of mercury, and there is no apparent reason why it should not do so, although the iron filings would in some cases have to be replaced by one of the agents mentioned above, or by some other decomposing agent. He has, however, had practically no experience on this point.

The process has been employed in his laboratory for five or six years, and the apparatus has been sent to mercury mines in Russia, China, Italy, and British Columbia, where it has been in constant use. At two mines, the gold cup process which was then in use has been abandoned in its favour on account of its greater convenience and speed, and at all these mines it has been constantly employed for valuing, selling, or buying the crude ore, for assaying tailings, and for controlling the working of the furnaces, etc. The above description may therefore be taken as that of a proved rather than a suggested method of assay.

DISCUSSION.

The PRESIDENT (Mr. Bevan) asked if the process could be used on a larger scale than Mr. Holloway had indicated, using a larger quantity of the ore.

Mr. HOLLOWAY said that the use of a larger quantity of ore did not increase the accuracy of the method if one had a good balance; in fact, the process worked better with the small quantities recommended in the paper.

Mr. KITTO said that for many years he had used a gold cup having a fairly large cavity, and had found no difficulty in getting very accurate results without the addition of the ingenious apparatus which Mr. Holloway had described. He had been much interested in the device for automatically cooling the silver foil. Probably where, as in a mine, a large number of determinations had to be made, it would be very useful, but under ordinary circumstances he really did not think it necessary to complicate the very simple original apparatus. He had had no difficulty in getting the flange of the gold cup firmly fixed and accurately adjusted to the edge of the crucible.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection, Determination, and Rate of Disappearance of Formaldehyde in Milk. R. H. Williams and H. C. Sherman. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1497.)—The authors find that by means of the potassium cyanide method (ANALYST, 1904, xxix., 5) formaldehyde in milk can be approximately determined at any concentration greater than 1 part in 160,000. They find that aqueous solutions of formaldehyde containing from 1 : 5000 to 1 : 40,000 lose steadily in strength on standing, the loss being not merely due to polymerization, but to actual destruction of the formaldehyde. Within the same limits, formaldehyde added to milk disappears about ten or twenty times as rapidly as from aqueous solutions of the same strength. Stronger solutions are much more stable; thus, an aqueous solution of 1 : 1,000 showed no appreciable loss after five months. From milk containing 1 part in 1,000 the rate of disappearance was the same as for an aqueous solution of 1 : 5,000. Souring of the milk does not affect the rate of disappearance. The hydrochloric acid and ferric chloride test is sensitive to a dilution of 1 : 250,000. Sourness of the milk does not affect the reaction. The test may be made more satisfactory by adding 50 to 75 c.c. of cold water to the milk after keeping it just below the boiling-point for one minute as usual; the colour obtained in this case, however, is very fugitive. The gallic acid test is much more delicate than the ferric chloride test. A. G. L.

Composition of the Fat of Pigs fed on Foods Rich in Oil. K. Farnsteiner, K. Lendrich, and P. Buttenberg. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 1-8.)—The particular object of the present experiments was to ascertain whether the oily constituents of the fodder were deposited in the body fat of pigs fed on such fodder. The results show that the fat of pigs fed on cottonseed-meal gives a decided reaction with Halphen's reagent, but that the fat in no case contains phytosterol, and is consequently thus distinguished from lard adulterated with cottonseed-oil. (*Cf.* ANALYST, 1904, p. 315.) W. P. S.

On the Occurrence of Arsenic in Wines. H. D. Gibbs and C. C. James. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1484.)—The authors have examined a total of 352 wines, some bottled and some in cask, and have found arsenic in 52 cases, in quantities ranging up to 1 part of arsenic in 2,500,000 of wine. Generally, however, the quantities found were much smaller than this. As probable sources of the arsenic they indicate the arsenical sprays used upon the vines, the sulphur burned for sulphuring the wine and receptacles, and, perhaps, also the lead-shot used in cleaning the bottles. The method used in testing for arsenic consisted in making the wine alkaline with milk of lime, evaporating to dryness, and igniting the residue at a low red heat. The ash was treated with sulphuric acid (1 to 3), the solution filtered and introduced into the generator of a Marsh apparatus. As generator a 250 c.c. Florence flask was used, Kahlbaum's zinc and pure sulphuric acid being used to generate the hydrogen. The action was started by adding a few drops of platinic chloride.* Immediately after adding the sulphuric acid the air in the apparatus was swept out by a rapid current of carbon dioxide, and the solution to be tested was then added. The evolved gas was passed through a spiral of lead acetate paper and then through calcium chloride, after which it entered a Jena glass tube of 0·7 cm. internal and 1·0 cm. external diameter drawn down to a constriction from 10 to 15 cm. long and 2 mm. external diameter. The tube was heated for a space of 10 to 15 cm., protected by wire gauze, before the constriction, the heating being continued for one hour. The electrolytic generator of Thorpe was also tried, but discarded because in some cases it failed to reveal the presence of any arsenic in wines known to contain arsenic.

A. G. L.

Process for Determining the Quality (Fineness) of Flour. N. Wender. (*Zeit. Untersuch. Nahr. Genussm.*, 1905, x., 747-756.)—Wheat and other grains contain an enzyme capable of liberating oxygen from hydrogen peroxide, and upon the fact that this enzyme resides principally in the embryo and outer portions of the grain, the author bases a process for distinguishing between best white flour and other flours containing more or less bran or inferior flour. It is recognised that, taken by itself, the colour of flour is no criterion in judging whether a sample is best quality, "seconds," "thirds," etc. The process is carried out as follows: 25 grams of the flour are rubbed down with 100 c.c. of water until free from lumps and then rinsed into a flask with a further 100 c.c. of water. The flask is closed by an indiarubber stopper carrying a tapped funnel and a delivery-tube connected to a measuring-tube filled with water. Ten c.c. of hydrogen peroxide are then introduced through the funnel, and the action allowed to proceed for about thirty minutes at a temperature of 20° C. The volume of the liberated gas is then read off. The following examples are given, in which the results are calculated for 100 grams of meal: Wheat starch, 8 c.c.; wheat flour, 169 c.c.; wheat bran, 342 c.c.; rye flour, 153 c.c.; rye bran, 330 c.c.; maize flour, 389 c.c. In a series of flours of decreasing quality, the finest flour gave 128 c.c., and the coarsest 486 c.c. The author admits that many more analyses must be made before any degrees of quality can be fixed by this method, but considers that the latter will be of use when the flours of different countries, mills, etc., have been examined and classified.

W. P. S.

A New Adulterant of Lemon-Juice. H. Matthes and F. Müller. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 20, 21.)—A sample of so-called lemon-juice gave the following results: Alcohol, by weight, 5·43 per cent.; total solids, 13·47 per cent.; total acidity, as citric acid, 8·19 per cent.; mineral matter, 0·205 per cent.; alkalinity of ash, 0·49 c.c. $\frac{N}{1}$ acid; phosphoric anhydride, 0·05 per cent.; polarization after distillation, $+4·5°$ in 100 mm. tube. The sample contained starch-syrup, evidently

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* It has been shown that the use, in this way, of platinic chloride is inimical to the correct estimation of arsenic. *Cf.* Chapman, this vol., p. 3.—EDITOR.

added to imitate the pectinous substances of pure lemon-juice and the amount of phosphoric anhydride found was probably due to the addition of a salt of this acid.
W. P. S.

Composition of Lemonade Essences. Utz. (*Zeit. offentl. Chem.*, 1906, xii., 12-13.)—The liquids to which the following analyses refer are sold for the purpose of making lemonade and similar drinks by diluting the liquid with water, soda-water, etc. They consist essentially of an aqueous solution of tartaric or citric acid, to which an alcoholic solution of certain flavouring substances (esters) are added. The figures express grams per 100 c.c.:

	Raspberry Essence A.	Raspberry Essence B.	Lemon Essence A.	Lemon Essence B.
Alcohol	14·55	19·40	19·91	27·84
Total solids	10·11	11·60	18·54	9·74
Mineral matter	0·065	0·174	0·082	0·119
Total acidity, as tartaric	8·029	11·56	15·57	9·38
Sugar...	none	none	none	none

All four samples were strongly coloured with coal-tar dyes, and two of them—Raspberry Essence B and Lemon Essence B—contained not inconsiderable quantities of saponin.
W. P. S.

On the Colouring Matter of Saffron. F. Decker. (*Chem. Zeit.*, 1906, xxx., 18.)—Crocetin, the colouring matter of saffron, has hitherto only been isolated in an amorphous form, since the readiness with which it resinified prevented its being obtained in a crystalline condition. The author, however, has succeeded in obtaining crocetin crystals of constant composition by digesting the resin-free solution of the colouring matter in very dilute sodium hydroxide solution at 60° to 70° C., with an excess of ammonium carbonate solution. The ammonium compound of crocetin was deposited from the liquid on cooling, in the form of yellow needles, which dissolved readily in alkalies, but were only sparingly in water and alcohol. The addition of ammonium carbonate to any of these solutions reprecipitated quantitatively the crocetin compound. An elementary analysis gave the following results: Carbon, 64·60; hydrogen, 8·70; nitrogen, 8·06; and oxygen, 18·64 per cent.
C. A. M.

Separation of the Conium Alkaloids. J. von Braun. (*Berichte*, 1905, xxxviii., 3108-3113; through *Pharm. Jour.*, 1905, vol. 75, 909.)—The separation of the alkaloids of the hemlock—namely, coniine, methyl-coniine, γ-coniceine, conhydrine, and pseudo-conhydrine, may be effected as follows: The greater portion of the coniine, which forms the chief constituent, is separated by distillation and the residue then fractionated up to 190° C., whereby the conhydrine (m.p. 118° C.) remains as an undistilled residue. The lower fractions are next benzoylated in alkaline solution, and the resulting oil, after dissolving in ether, is extracted with acid to remove the methyl-coniine. The ethereal solution is then concentrated and treated with petroleum spirit (ligroin) to precipitate the benzoyl-amino-butyl-propyl-ketone, which is formed by the action of benzoyl chloride on γ-coniceine. The mother-liquor is redistilled and yields benzoyl coniine (b.p. 203° to 204° C. at 16 mm. pressure), and some more of the benzoyl-amino-ketone. Starting with 104 grams of material, the author was able to obtain 1 gram of conhydrine, 7 grams of methyl-coniine, 52 grams of amino-ketone corresponding with 26 grams of γ-coniceine, and 124 grams of benzoyl-coniine, corresponding with 68 grams of coniine, total 102 grams. The coniine recovered from the benzoyl derivative proved to be a mixture consisting of *l*-coniine and a little *d*-coniine. The substance described above as methyl-coniine has $[\alpha]_D$ 35·66 at 24° C., and appears to be a simple substance. It yields a picrate melting at 114° C., a platinichloride melting at 195° C., and an aurichloride between 80° and 90° C. W. P. S.

The Oleoresin of Pinus Longifolia. F. Rabak. (*Pharm. Rev.*, 1905, xxiii., 229; through *Pharm. Jour.*, 1906, vol. 76, 11.)—The oleo-resin of *Pinus longifolia*, a native of the lower Himalayas, is a white, opaque, tough substance showing a partially crystalline structure. Its odour is lemon-like. A sample examined had a specific gravity of 0·866 and $[\alpha]_D + 2° 48'$. It yielded 18·5 per cent. of essential oil on distillation, the oil having a characteristic pinene odour, and a faint aroma of limonene. On fractionation, dextropinene and lævolimonene were isolated. The residual resin was whitish and brittle; it had an acid value of 142 and ester value of 13. When dissolved in acetic acid the solution deposited crystals of a resin acid melting at 138° to 140° C. W. P. S.

A Distinctive Reaction for Euphorbium Resin. A. Tschirch and Paul. (*Pharm. Zeit.*, 1905, l., 561; through *Pharm. Journ.*, 1906, vol. 76, 35.)—About 0·01 gram of the resin is treated with 10 c.c. of petroleum spirit, and the mixture filtered. The filtrate is allowed to flow on to the surface of 20 c.c. of sulphuric acid, to which 1 drop of nitric acid has been added. A red-coloured zone is obtained at the junction of the two liquids; on shaking, the acid layer becomes red and, in the course of a day or two, turns brown. W. P. S.

Estimation of Formaldehyde in Formaldehyde Pastilles (Trioxymethylene). Ernst Rüst. (*Zeit. angew. Chem.*, xix., 138.)—The author claims the following method of estimating formaldehyde to be superior to the older ones: Two grams powdered trioxymethylene are added to a 250 c.c. Erlenmeyer flask, in the neck of which is fitted a funnel. The solid is washed in with 70 c.c. normal sodium hydroxide solution. To this mixture 9 to 10 grams of 30 per cent. hydrogen peroxide is added slowly at first to prevent undue heating. After standing for two hours the mixture is warmed gently to boiling, to decompose the excess of hydrogen peroxide. The funnel, after being carefully washed with water, is removed, a small excess of normal sulphuric acid solution added, and the mixture titrated back with normal sodium hydroxide solution, using phenolphthalein as indicator. One c.c. of normal sodium hydroxide corresponds to 0·03 gram CH_2O. The original substance must be tested for alkalies or acids before this method is applied, and these must be taken into account in the final calculation. H. D. L.

The Detection of Iodine Compounds in the Dry Way. B. Merk. (*Pharm. Zeit.*, 1905, l., 1022; through *Chem. Zeit. Rep.*, 1905, xxix., 405.)—The substance under examination is rubbed with a little potassium persulphate and some soluble starch, the mixture becoming more or less blue in the presence of iodine compounds not containing oxygen:

$$2KI + K_2S_2O_8 = 2K_2SO_4 + I_2.$$

C. A. M.

TOXICOLOGICAL ANALYSIS.

Toxicology of Atropine. C. Ipsen-Innsbruck. (*Zeit. angew. Chem.*, xix., 141.)—The author has investigated the stability of atropine in the presence of decomposing organic matter, the method of absorption of this alkaloid in, and excretion from, living organisms. The results, which are opposed to those of previous investigations, are summarized in the following manner: Atropine is rapidly absorbed by all parts of the body and distributed in the blood. The separation rapidly follows from all the natural outlets, lasting from three to fourteen days, depending on the animal. Further, 0·03 gram atropine, after standing for twelve years in contact with decomposing blood, beer, and other organic bodies, can still be detected. H. D. L.

Toxicity of Eggs when administered Hypodermically. G. Loisel. (*Comp. Rend.*, 1905, cxli., 730; through *Pharm. Journ.*, 1906, vol. 76, 65.)—Eggs are found to be by no means free from toxicity when administered hypodermically. The lethal dose for rabbits of the emulsion of yolk of ducks' eggs with water is 10 c.c. per kilo of body-weight. A 10 per cent. solution of the dried yolk in saline water is similarly toxic by intravenous injection in doses of 80 c.c. Hens' eggs are slightly less toxic, and turtles' eggs from Mauritius somewhat more so. In fact, all three are markedly poisonous when injected either into the veins, or hypodermically, or into the peritoneal cavity. The symptoms which precede death indicate that the nervous system is profoundly affected by the poison. W. P. S.

ORGANIC ANALYSIS.

Preparation of Pure Ethyl Alcohol. L. W. Winkler. (*Berichte*, 1905, xxxviii., 3612-3616; through *Pharm. Journ.*, 1905, vol. 76, 12.)—Acetaldehyde is removed from commercial absolute alcohol by allowing the sample to stand over finely-powdered silver oxide for several days, a little potassium hydroxide being also added. The amount of silver oxide required is never more than a few grams per litre, and about 1·2 grams of potassium hydroxide is sufficient to neutralize all the acetic acid resulting from the oxidation of the aldehyde. The mixture should be shaken from time to time. The water present in the alcohol is then removed by heating the latter for several hours over, and finally distilling from, metallic calcium filings, using about 20 grams of the metal per litre. The alcohol so obtained can be once more distilled from metallic calcium. The advantage in the use of calcium over sodium lies in the fact that calcium hydroxide is totally insoluble in alcohol, and therefore as long as the alcohol contains moisture a white precipitate will be formed.

The author has made a number of determinations of the specific gravity of pure ethyl alcohol, and has deduced from them the following formula for calculating the specific gravity of alcohol at any temperature from 0° to 30° C.:

$$\text{Specific gravity } 0^\circ\text{–}30^\circ = 0{\cdot}80629 - 0{\cdot}000838t - 0{\cdot}0000004t^2,$$

where 0·80629 is the specific gravity of alcohol *in vacuo* compared with that of water at 4° C. The boiling-point of pure alcohol was found to be 78·37° C. at 760 mm. pressure. It was observed that absolute alcohol is not so hygroscopic as is commonly imagined; 200 c.c. of absolute alcohol absorbed less than 0·1 per cent. of water after fifteen minutes' exposure to the air. W. P. S.

The Determination of Traces of Benzaldehyde. H. Hérissey. (*Journ. Pharm. Chim.*, 1906, xxiii., 60-65.)—Phenylhydrazine reacts with benzaldehyde to form an insoluble phenylhydrazone—

$$C_6H_5NH.NH_2 + C_6H_5.COH = C_6H_5.CH.N.NH.C_6H_5 + H_2O.$$

The reagent used by the author consisted of 1 c.c. of freshly distilled phenylhydrazine and 0·5 c.c. of glacial acetic acid in 100 c.c. of water. On treating an aqueous solution of benzaldehyde with an equal volume of this reagent, and allowing the mixture to stand for twenty-four hours at 15° to 20° C., there was formed an insoluble crystalline precipitate, which could be collected in a Gooch's crucible, washed with a known quantity of cold water, and dried in a vacuum over sulphuric acid until constant in weight. It was found, however, that very faint traces of the aldehyde required a very long time for complete combination, and, on the other hand, the results were liable to be too high if the reagent was allowed to act for several days. But by heating the mixture on the boiling water-bath for twenty minutes, and then allowing the liquid to stand for twelve hours before filtration, washing the precipitate in the Gooch's crucible with 20 c.c. of water and drying it *in vacuo*, excellent results were obtained, especially when the quantity of phenylhydrazone did not exceed 0·1 to 0·25 gram. C. A. M.

On the Determination of Sugar with Fehling's Solution. F. P. Lavalle. (*Chem. Zeit.*, 1906, xxx., 17.)—It is often difficult to determine the end-point of the reaction in titrating a solution of glucose, owing to the cuprous oxide subsiding but slowly, and reflecting its colour to the surface. The author obviates this by adding an excess of alkali, and thus preventing entirely the separation of the cuprous oxide. From 5 to 10 c.c. of Fehling's solution are placed in a 200 c.c. porcelain basin, together with 30 c.c. of sodium hydroxide solution (1:3) and 50 to 60 c.c. of water, and the liquid heated to incipient boiling, and then titrated with the sugar solution, the reaction being complete as soon as the last drop causes the blue colour of the Fehling's solution to disappear. Test experiments are stated to have given very satisfactory results. C. A. M.

The Detection and Determination of Trehalose by Means of Trehalase. P. Harang. (*Journ. Pharm. Chim.*, 1906, xxiii., 16-20.)—The method is based upon the fact that trehalose ($\alpha = +197^\circ\ 3'$) is inverted by the specific enzyme trehalase into two molecules of glucose, which will, of course, reduce Fehling's solution. Thus, if a 1 per cent. aqueous solution of anhydrous trehalose giving in the polarimeter a reading of $\alpha = +3^\circ\ 57'$ be treated with trehalase, the liquid, after complete inversion of the hexabiose, will be found by means of Fehling's solution to contain a quantity of reducing sugar corresponding to 1·052 grams of glucose, and the deviation ($l = 2$) will be $+1^\circ\ 6'$, a difference of $2^\circ\ 51'$. If, then, the difference between the rotations calculated from the amount of glucose formed under the action of the enzyme coincides with the polarimetric readings, the presence of trehalose may be inferred, and its amount may be calculated, provided that the enzymic action is complete.

In order to obtain the specific enzyme trehalase the author incubates a cultivation of *Aspergillus niger* on Raulin's fluid at 33° C. until the first signs of fructification (about twenty-four hours). The nutrient fluid is then decanted and replaced with water, and the cultivation allowed to stand at the ordinary temperature for five or six days until fructification appears, the water being renewed every twenty-four hours. The mould fungus is then pressed between filter-papers, finely divided, left for three hours in contact with a fourth of its weight of 95 per cent. alcohol, drained with the aid of a filter-pump, and finally dried in the oven at 33° C. and powdered. This preparation has a strong enzymic action upon trehalose, 0·5 gram being sufficient to invert 1 gram of the sugar in 100 c.c. of water within forty-eight hours at 33° C.

Before using the enzyme for the detection of trehalose in mushrooms, etc., it is necessary to eliminate substances such as glycogen, which can also be attacked by it. The fungi are twice extracted, as soon as possible after being gathered, for ten minutes with their own weight of boiling 80 per cent. alcohol, and the united extracts filtered. The filtrate is distilled under reduced pressure until the residue is only 100 c.c. (from 1 kilo of fresh mushrooms). On now adding 4 parts by volume of 80 per cent. alcohol to the residue there is an abundant precipitate, which is left to subside until the next day. The clear liquid is then decanted, and the residue mixed with a few c.c. of water, taken up with 4 parts by volume of 80 per cent. alcohol, and boiled for twenty minutes on the water-bath under a reflux condenser. It is then allowed to cool and the supernatant liquid decanted, united with the washings, and evaporated to dryness *in vacuo*. Finally, the residue is taken up with water saturated with thymol (100 c.c. for each 200 grams of mushrooms), and this solution treated with enzyme. C. A. M.

The Characteristics of Certain Animal Fats. C. Schneider and S. Blumenfeld. (*Chem. Zeit.*, 1905, xxx., 53, 54.)—The following physical and chemical values were obtained :

Animal.	Fats.							Fatty Acids.						
	Specific Gravity at 15° C.	Refractometer.	Acid Value.	Saponification Value.	Iodine Value (Hübl).	Hehner Value.	Reichert-Meissl Value.	Specific Gravity at 15° C.	Refractometer.	Melting-point. ° C.	Solidification-point, ° C.	Neutralization Value.	Iodine Value.	Acetyl Value.
Vikare seal (*Phoca fœtida*)	0·9321	87·0 at 20° C.	0·48	188·5	191·35	95·6	1·55	0·9172	74·1 at 20° C.	14·0	13·0–14·0	198·0	195·3	—
Vikare seal (*Phoca fœtida*)	0·9336	—	1·08	189·0	193·3	95·8	0·96	—	—	—	13·0–14·0	196·0	201·8	—
Porpoise (*Phocæna communis*)	0·9334	62·7 at 25° C.	1·2	224·8	111·2	85·5	42·1	0·9121	54·3 at 25° C.	—	18·0	207·0	126·0	4·55
Coot (*Fulica atra*)	0·9163	62·9 at 25° C.	1·66	192·6	87·13	95·2	0·35	0·9151	44·7 at 35° C.	33·5–34·5	30·5	—	84·8	—
Crane (*Grus cinerea*)	0·9222	61·5 at 20° C.	9·33	191·2	75·25	95·7	0·13	0·9005	40·8 at 30° C.	31·0	29·3	201·0	73·5	1·35
Lynx (*Lynx europæus*)	0·9248	70·0 at 20° C.	0·81	190·22	110·6	95·8	0·43	0·9412	53·9 at 35° C.	35·5	35·0	202·7	111·8	7·67
Glutton (*Gulo borealis*) body fat	0·9153	54·2 at 30° C.	5·84	193·3	54·36	95·4	0·12	0·9118	31·9 at 45° C.	40·0–41·0	37·5	203·4	55·5	3·0
Glutton (*Gulo borealis*) kidney fat	0·9230	45·2 at 45° C.	—	193·3	50·82	95·8	—	—	31·7 at 45° C.	40·0–41·0	—	203·0	52·8	—
Bear (*Ursa arctos*)	0·9156	60·8 at 20° C.	30·6	191·0	80·7	94·5	0·33	0·9347	43·0 at 40° C.	37·5	36·1	203·0	76·5	5·7

The high Reichert value of the porpoise fat was due to the presence of a considerable quantity of the glycerides of valeric acid. The fat, which was derived from the body, was oily, and had a pale yellow colour. Of the other fats examined, that of the seal was a soft yellow semi-solid; the fats of the coot, crane, and lynx were soft white or light yellow semi-solid masses; the fat of the glutton was a white, not very hard fat; and the bear's fat had the consistency of vaseline at 15° C. It is pointed out that the iodine value of the fat of the lynx is the highest yet recorded for land beasts of prey. C. A. M.

Determination of the Total Organic Acids in Tobacco. J. Tóth. (*Chem. Zeit.*, 1906, xxx., 57, 58.)—The method proposed is similar to the one described previously by the author for the determination of nicotine (ANALYST, 1902, p. 12). Two grams of the dry powdered tobacco are moistened with 2·5 c.c. of 20 per cent. sulphuric acid, and then mixed with sufficient plaster of Paris to form a dry powder. This is placed in a well-corked cylinder and shaken for forty-eight hours with 100 c.c. of anhydrous ether; 50 c.c. of the ethereal solution are then drawn off, evaporated slowly, and the residue dissolved in warm water. The solution is now titrated with $\frac{N}{2}$ sodium hydroxide solution, using alkanna tincture as indicator (ANALYST, 1903, p. 193), and the result calculated into anhydrous oxalic acid. In thirty-two samples of various tobaccos examined, the writer found from 3·6 to 8·7 per cent. of total organic acids (expressed as oxalic acid), and the quantity of organic acid was found to be proportional to the burning quality of the several tobaccos—that is, bad-burning tobaccos contained the most acid. W. P. S.

Estimation of Tannin in Crude Products. A. Manea. (*Chem. Zeit. Rep.*, 1905, xxix., 381.)—The principle of the method lies in the fact that when a certain quantity of a tannin solution is added to a mixture of a known quantity of glacial acetic acid and neutral lead acetate of known dilution, the tannin alone is precipitated with the lead, whilst all other substances remain in solution. The author has obtained results which give not more than 0·5 per cent. difference in analyses of the same raw material. E. K. H.

Estimation of Caoutchouc. T. Budde. (*Chem. Zeit. Rep.*, 1905, xxix., 393.)—The process recommended, after many experiments, is as follows: A solution of bromine in carbon tetrachloride is used as a brominating liquid, iodine being added as a halogen carrier, the action being then continuous. The proportions used by the author are: 16 grams bromine (6 c.c.) and 1 gram iodine dissolved in carbon tetrachloride and made up to 1 litre. For the estimation about 1 gram of the sample of caoutchouc is placed in a 100 c.c. flask, covered with carbon tetrachloride, and allowed to stand, with frequent shaking, until the caoutchouc has dissolved, or is evenly divided. The flask is then filled to the mark and well shaken. Ten c.c. are taken for each analysis. This is filtered, if necessary, in an Allihn's tube and washed with about 50 c.c. CCl_4. (If the solution is too viscous, it must be diluted.) To the filtrate 50 c.c. of the brominating solution are added, when a jelly-like substance is soon precipitated. After the solution has become clear (quarter of an hour), half its volume of alcohol is added; the liquid is now clear yellow, and the tetrabrom-caoutchouc is transformed into the white form. It can now be easily transferred to a weighed filter paper, washed first with a mixture of 2 parts carbon tetrachloride and 1 part alcohol, and then with pure alcohol, and dried to constant weight at 60° C.; 456 grams of tetrabrom-caoutchouc = 136 grams of pure caoutchouc. Caoutchouc resin and oxidized caoutchoucs do not take part in the bromine reaction. The process recommends itself by its simplicity, and especially by the fact that it gives the true content of pure caoutchouc. E. K. H.

The Direct Determination of the Acetyl and Benzoyl Groups. R. Meyer and E. Hartmann. (*Berichte*, 1905, xxxviii., 3956.)—From 0·5 to 0·7 gram of the substance is heated with about 5 grams of pure sodium hydroxide and as small a quantity as possible of methyl alcohol which has been purified by distillation with potassium hydroxide. After about an hour the saponification is complete in the case of most acetates, and the solution is then mixed with 50 c.c. of phosphoric acid (specific gravity 1·104), and distilled in a current of steam, a little lime being placed in the vessel in which the steam is generated. In order to determine whether the distillation is complete, portions of the distillate are titrated from time to time after it is thought that the bulk of the acid has passed over. The distillation should be continued until 150 c.c. of the distillate consume not more than 1 or 2 drops of $\frac{N}{10}$ baryta solution, phenolphthalein being used as indicator. The determination of the benzoyl group is carried out in the same way, and gives equally satisfactory results. C. A. M.

INORGANIC ANALYSIS.

Provisional Methods for Copper, Lead, and Zinc of the Committee on Uniformity in Technical Analysis of the Western Association of Technical Chemists and Metallurgists. (*Chem. Engineer*, 1905, ii., 287.)

Copper.—The iodometric method is used, the sodium thiosulphate solution (20 grams per litre) being standardized by dissolving 0·2 to 0·5 gram of copper foil in 5 c.c. of nitric acid and evaporating to 2 or 3 c.c.; after adding 5 c.c. of hot water and 6 c.c. of ammonia, the whole is boiled for a few minutes, cooled, and diluted to 75 or 80 c.c. After adding 8 c.c. of acetic acid and 10 c.c. of potassium iodide solution (30 per cent.), and well shaking, the liquid is titrated with the thiosulphate, 4 or 5 c.c. of starch solution (1 gram per 200 c.c.) being added after the colour has been changed from brown to yellow. To determine copper in ores, 0·5 gram of the sample is heated with 2 c.c. of nitric acid, 3 c.c. of hydrochloric acid, and 4 c.c. of sulphuric acid, until white fumes are evolved. After cooling and diluting with 25 c.c. of water, the whole is boiled with a piece of sheet aluminium; 10 c.c. of hydrogen sulphide water are then added to insure complete precipitation, and the precipitate filtered off, washing three times by decantation; 55 c.c. strong nitric acid are then poured over the copper and aluminium; the aluminium is removed and washed with a little hot water, and the filter washed with strong bromine water. The solution is then evaporated down to 2 or 3 c.c.; 5 c.c. of hot water and 6 c.c. of ammonia are added; the whole is boiled, acidulated with 8 c.c. of acetic acid, and titrated as above.

Lead.—The ammonium molybdate method is used. To standardize the solution, 0·3 to 0·5 gram of lead foil is dissolved in 10 to 15 c.c. of 1·2 nitric acid; 20 c.c. of 1·1 sulphuric acid are then added; the whole is well stirred and allowed to settle. The liquid is then decanted off through a filter, and the precipitate washed three or four times with 2 per cent. sulphuric acid, and finally once with cold water. The filter is then washed free from lead sulphate by repeatedly passing 50 c.c. of a hot 20 per cent. ammonium acetate solution through it, and finally washing with hot water. The solution is then heated with the main bulk of the precipitate until the latter is dissolved, when it is diluted to 200 c.c. made just acid with acetic acid, and titrated with ammonium molybdate solution (8·64 grams per litre) until a drop of the liquid gives a yellow colour with a drop of tannic acid solution (1 gram in 300 c.c. water). For ores, 0·5 to 1 gram is heated with 10 c.c. of strong nitric acid and 10 c.c. of strong sulphuric acid until white fumes appear, when the liquid is cooled, diluted to 50 c.c., boiled, and treated as above.

Zinc.—The ferrocyanide method is used. To standardize the solution 0·2 to 0·3 gram of freshly-ignited zinc oxide is dissolved in 10 c.c. of hydrochloric acid, and neutralized with ammonia; 6 c.c. of hydrochloric acid are then added; the liquid is diluted to 180 c.c. with water, boiled, and titrated as follows: The solution is divided into two equal parts; one of these is titrated quickly, after which the other part is added, and the titration finished by adding nearly enough of the ferrocyanide solution at once to precipitate the zinc, and then very carefully adding the solution until a yellow colour is obtained with a drop of uranium acetate solution containing 4·5 grams in 100 c.c. of water. The ferrocyanide solution contains 21·6 grams per litre. For ores, 0·5 to 1 gram is dissolved in 4 to 8 c.c. of hydrochloric acid, and an equal amount of nitric acid. The solution is evaporated down to one-third its volume; any gelatinous silica separated is filtered off, and the filtrate evaporated to dryness with 15 c.c. of a saturated solution of potassium chlorate in nitric acid. Without any baking, the residue is boiled with 40 c.c. of a solution made by dissolving 60 grams of ammonium chloride in 250 c.c. of water and 150 c.c. of concentrated ammonia. The precipitate obtained is filtered off and washed with the same ammonium chloride solution; if much iron is present, the precipitate is dissolved in the minimum of hydrochloric acid, and reprecipitated with the ammonium chloride solution. The filtrate is neutralized, 15 c.c. of hydrochloric acid and 15 grams of granulated lead are added, and the whole is boiled until all the copper is precipitated, after which the titration is made as above. All the conditions should be the same in all titrations, as far as possible. The temperature should always be near the boiling-point. A. G. L.

The Volumetric Determination of Lead as Iodate. L. Moser. (*Chem. Zeit.*, 1906, xxx., 9.)—On treating a solution of a lead salt with potassium iodate in moderate excess, there is formed a heavy crystalline precipitate of lead iodate, which is nearly insoluble in water, and only sparingly soluble in nitric acid, but dissolves readily in hydrochloric acid with the formation of chlorine. This lead iodate is much more insoluble than the sulphate, and good results can be obtained in analytical determinations based on the reaction. The solution of the lead salt, which may contain acetic acid or a little nitric acid, is treated with a measured quantity of standard potassium iodate solution, made up to a definite volume, and shaken, and the lead iodate allowed to subside. An aliquot portion of the clear supernatant liquid is then titrated with thiosulphate solution, after the addition of potassium iodide and dilute sulphuric acid. The excess of iodate thus determined is subtracted from that originally added, and the difference corresponds to the amount of lead present, 2 parts of potassium iodate being equivalent to 1 part of lead.

In examining commercial sugar of lead, 1 gram of the sample is dissolved in water, mixed with 70 c.c. of decinormal potassium iodate solution, and made up to 200 c.c. An aliquot portion (50 c.c.) of the clear supernatant liquid is then treated with potassium iodide, and the liberated iodine titrated with standard thiosulphate solution. The amount of iodine found corresponds to the amount of free acid in the sugar of lead. Sulphuric acid is now added, and the main quantity of iodine titrated. When the original sample does not yield a clear solution, a few drops of acetic acid are added to dissolve basic salts, etc. C. A. M.

The Rapid Precipitation of Antimony Electrolytically. Julia Langness and Edgar F. Smith (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1524.)—In consequence of Fischer and Boddaert's objections (*Zeits. f. Elektrochemie*, x. 950), the authors have repeated Exner's work on the deposition of antimony, using the rotating anode (ANALYST, 1903, xxviii., 330), and they find the method to give excellent results. They used an almost boiling solution of a total volume of 70 c.c., containing 0·24 gram of antimony in either state of oxidation, as well as 15 c.c. of sodium sulphide solution (specific gravity, 1·18), 3 grams of potassium cyanide, and 1 c.c. of 10 per cent. sodium hydroxide. Only 2 grams of potassium cyanide need be added if only 10 c.c. of sodium sulphide solution are present. The current was 6 ampères per 100 sq. cm. at 3·5 to 4 volts, the time being fifteen minutes. The method may be applied to the analysis of stibnite, the ore being directly treated with sodium sulphide, and to the separation of arsenic from antimony. In six experiments in which 0·1268 gram of antimony and 0·2000 gram of arsenic were present, the maximum error on the antimony was 0·0001 gram. A. G. L.

The Determination of Bismuth and its Separation from other Heavy Metals. H. Salkowski. (*Berichte*, 1905, xxxviii., 3943, 3944.)—It was suggested by the author in 1868 that it would be preferable to determine bismuth in the form of a phosphate rather than as an arsenate, and he has now worked out the details of the method. The precipitate can be ignited without fear of reduction and weighed as $BiPO_4$. It is essential that the solution should not contain hydrochloric acid or chlorides, and that free nitric acid should not be present in any considerable quantity. Good results are stated to have been obtained in this way in the separation of bismuth from copper and lead, but the details are not described. C. A. M.

The Iodometric Determination of Copper. T. Brown, Jun. (*Chem. Engineer*, 1905, ii., 307.)—The method described consists in dissolving from 2·5 to 0·5 gram of the ore in *aqua regia*, evaporating the solution with sulphuric acid, filtering from gangue and lead sulphate, and precipitating the filtrate with sodium thiosulphate. In precipitating with thiosulphate the author recommends boiling the liquid for one half hour for high-grade ores, and for one hour or even longer for low-grade ores; if too much evaporation takes place, water should be added. The precipitate is filtered off, washed with hot water, and roasted to oxide at a dull red heat. The copper oxide obtained may contain antimony, bismuth, and silver. It is placed in an 8-ounce flask, nitric acid, water, and about 0·5 gram of potassium chlorate are added, and the whole is boiled down to about half its original volume. Should any chlorate become decomposed a little water is added, and the chlorine liberated boiled off. After cooling, a little water and a slight excess of ammonia are added, and the whole is boiled again for a few minutes; this treatment insures a more distinct end-point. The solution is then acidified with acetic acid, the determination finished as usual with potassium iodide and standard thiosulphate solution.

A. G. L.

Analysis of Tungsten-Tin Minerals. Henri Angenot. (*Zeit. angew. Chem.*, xix., 140.)—On account of the extreme difficulty experienced in the estimation of stannic oxide and tungstic acid in presence of each other, the author has introduced the following modification of the Bornträger method: One gram of the finely powdered mineral is carefully fused with sodium peroxide in an iron crucible for a quarter of an hour. This is digested with water and made up to 250 c.c. If lead be present, it must be separated with carbon dioxide and the mixture finally filtered. One hundred c.c. of the filtrate are treated with a mixture of 15 c.c. nitric acid and 45 c.c. hydrochloric acid, and then evaporated to dryness. The residue is dissolved in a mixture of 100 grams ammonium chloride, 100 grams hydrochloric acid, and 1,000 c.c. water, and filtered. The undissolved tungstic acid is dissolved in warm ammonia, and the process repeated, and the insoluble product finally ignited.

A second 100 c.c. is used for the estimation of the tin. Two to three grams of zinc are added, and the whole allowed to stand for one hour at 60° C., then filtered from the precipitated oxide of tungsten, and the filtrate treated with sulphuretted hydrogen to remove the tin, which is weighed as sulphide. H. D. L.

The Separation of Iron from Zinc. V. Komar. (*Chem. Zeit.*, 1906, xxx., 31, 32.)—The zinc and iron salts are converted into sulphates, dissolved in sulphuric acid (specific gravity, 1·3), and the solution oxidized by the addition of a little nitric acid. The mixture is then evaporated, at first on the water-bath, then on the sand-bath, and the residue finally heated over a free flame, care being taken to avoid crepitation. After igniting at a dull-red heat, the residue is dissolved in water and the ferric oxide collected on a filter, washed, and weighed, Zinc sulphate does not decompose at a temperature of 700° C., or more. W. P. S.

Standardizing Potassium Permanganate for Iron Determinations. Richard K. Meade. (*Chem. Engineer*, 1905, ii., 281.)—As a standard for permanganate solutions, the author prefers to use pure electrolytic iron. This is obtained by electrolysing, in a glass beaker, a solution containing 0·5 gram of iron as pure ferrous or ammonium ferrous sulphate dissolved in 250 c.c. of saturated ammonium oxalate solution. The anode is formed of a centrally-placed piece of platinum wire, the cathode consisting of two pieces of platinum foil 1½ inches square. After electrolysing for two hours with a current of from 0·5 to 0·7 ampère, about 0·15 gram of iron will be deposited on each piece of foil. After weighing each piece is dissolved separately in a mixture of 500 c.c. water and 50 c.c. of sulphuric acid, which has been previously boiled for some time, whilst a current of carbon dioxide was led through it. The flask and the dissolved iron are allowed to cool in the current of carbon dioxide, and titrated as usual. Proceeding in this way, it is found that the iron value of apparently pure iron wire is often above 100 per cent. A. G. L.

The Use of Ammonium Persulphate in the Determination of Chromium in Steel. Harry E. Walters. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1551; and *Proc. Eng. Soc. West Pennsylvania*, 1905, xxi., 465.)—The method consists in determining chromium and manganese together, after oxidation by ammonium persulphate, by means of ferrous sulphate, and determining the manganese separately by means of sodium arsenite: 1·25 grams of the sample are dissolved in 35 c.c. dilute sulphuric acid (1 : 5), a small amount of ammonium persulphate being added to oxidize the iron and carbonaceous matter; the liquid is diluted to 100 c.c., 40 c.c. of silver nitrate solution (containing 0·16 gram of the salt), and then 5 to 7 grams of slightly moist ammonium persulphate are added, and the whole is boiled for five minutes, after which the liquid is cooled and made up to 500 c.c. On 100 c.c. of this solution the manganese is then determined by titrating in a porcelain dish with sodium arsenite solution, made up according to Blair, and standardized against a chrome steel of known manganese content. To the remaining 400 c.c. a measured volume of ferrous sulphate solution (22·5 grams of ferrous sulphate and 50 c.c. of strong sulphuric acid per litre) is added, and the excess of ferrous sulphate determined with standard permanganate solution (1·82 grams of the salt per litre). The latter may be standardized against iron or against a pure chromium salt; it should be compared with the ferrous sulphate solution every day. Test results obtained in this way on pure solutions containing iron, manganese, and chromium, and on chromium steels, are excellent for both the manganese and chromium. If tungsten or molybdenum is also present, a carbide of chromium insoluble in sulphuric acid, may be left, which requires fusion with sodium carbonate. If vanadium is present, the manganese cannot be determined by sodium arsenite; in this case the bismuthate or Ford's method may be used. A. G. L.

Estimation of Chromium and Manganese. A. Kleine. (*Chem. Zeit. Rep.*, 1905, xxix., 380.)—Five grams of steel shavings are dissolved in 500 c.c. HCl (1·12) by the aid of heat, the solution is heated to boiling, and all subchlorides converted into perchlorides by adding 3 c.c. of HNO_3 (1·4) drop by drop. The solution is then evaporated to a syrup, and the iron extracted by shaking with ether. The iron-free solution is evaporated to dryness, the residue taken up by 10 c.c. sulphuric acid (1 : 10), the solution diluted, treated with ammonium persulphate (60 grams per litre), and boiled for fifteen to twenty minutes. The precipitated manganese peroxide is filtered off and washed. The chromium is estimated in the filtrate by adding excess of iron sulphate solution (50 grams $FeSO_4$ in 800 c.c. $H_2O + 200$ c.c. H_2SO_4), and the excess titrated with standard permanganate solution. The iron titre of the permanganate ×0·31 gives the chromium titre. The washed manganese precipitate is then transferred to the precipitation flask, 10 c.c. of sulphuric acid added, and sufficient oxalic acid solution run in to completely dissolve the precipitate. The excess of oxalic acid, after dilution with 200 to 300 c.c. of hot water, is estimated by permanganate. To obtain the manganese titre of the permanganate the iron titre must be multiplied by 0·501 (according to Ledebur), and not by the older value 0·491.

For crude iron and difficultly soluble chromium steel 5 grams of the metal should be dissolved in 50 c.c. of $HCl + 5$ c.c. HNO_3; the solution is then diluted, filtered from silica, evaporated and shaken up with ether, and treated as above. For steel with a high percentage of chromium the filtrate from the manganese precipitate should be made up to 500 c.c. and the chromium estimated in an aliquot part. E. K. H.

The Ford-Williams Method for High-grade Manganese Ores. J. James Skinner. (*Chem. Engineer*, 1905, ii., 279.)—0·5 gram of the ore is digested with 20 c.c. hydrochloric acid until the residue is white, after which the solution is evaporated down to a syrup. It is then boiled with 110 c.c. nitric acid until the red fumes are almost wholly driven off, when half a teaspoonful of fine asbestos fibre is added carefully, and boiling continued for one minute more, when 8 grams potassium chlorate are added, and the whole boiled for another ten minutes. The precipitate is then filtered off, as usual, on an asbestos filter. Precipitate and filter are then dropped into a beaker containing 0·750 gram of pure oxalic acid, 50 c.c. of dilute sulphuric acid, and 150 c.c. of warm water. After gently warming until the whole of the manganese dioxide has dissolved, the liquid is titrated with standard potassium permanganate, with which another portion of 0·750 gram oxalic acid is also titrated, the difference corresponding to the manganese.

For ores containing manganese in a form insoluble in hydrochloric acid, the insoluble portion is filtered off, treated with hydrofluoric and sulphuric acids, and, after evaporating off the hydrofluoric acid, dissolved in hydrochloric acid and added to the main solution. A. G. L.

A New Reagent for Potassium. E. P. Alvarez. (*Gazz. Chim. Ital.*, 1905, xxxv., 463; through *Chem. Zeit. Rep.*, 1905, xxix., 404, 405.)—The reagent consists of a saturated (5 per cent.) solution of amido-naphthol sulphonic acid,

$$C_{10}H_5\begin{cases}NH_2(1)\\OH(2)\\SO_3Na(6).\end{cases}$$

It should be prepared with recently-boiled cold water shortly before it is required for

use. It can only be kept in black bottles from which the air is carefully excluded. The precipitate of potassium amido-naphthol-sulphonate is given by all potassium salts, including the iodide, provided the solution be neutral. Ammonium salts, and magnesium salts in the presence of ammonium chloride, are not precipitated by the reagent. Solutions of potassium salts of the strength of 5 to 10 per cent. give the reaction almost immediately; those containing from 3 to 5 per cent. do not give a precipitate until after ten minutes; and those containing 1 per cent. not until after several hours.
C. A. M.

Colorimetric Estimation of Selenium. J. E. Clenell. (*Chem. Zeit. Rep.*, 1905, xxix., 392.)—The selenium must be brought into solution as selenite or selenate; this can be effected by treatment with concentrated nitric acid and subsequent addition of hydrochloric acid; the free chlorine must then be driven off. A white precipitate in the solution is of no account, and may, if necessary, be produced in the comparison test by precipitating zinc chloride with potassium ferrocyanide. The solution is heated to boiling, and a few c.c. of a 5 per cent. solution of sodium bisulphite added. In case of cyanide solutions, 100 c.c. boiled with 10 c.c. HCl for five minutes give the precipitate of selenium without any addition of sulphite. The finely divided selenium remains some time in suspension, and the tint it gives is compared with that of known amounts of a standard solution prepared by shaking 0·1 gram selenium with strong bromine water until dissolved, adding bicarbonate gradually until a colourless solution is obtained, and making up to a litre. The standard tests are boiled with 5 to 10 c.c. of sodium bisulphite, as before.

The only element which interferes with this process is copper, which with potassium ferrocyanide gives a colour similar to that of the suspended selenium.

The estimation takes about fifteen minutes.
E. K. H.

The Use of the Rotating Anode and Mercury Cathode in Electro-analysis. Second Paper. Lily G. Kollock and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1527.)—Continuing their work, the authors have applied the rotating anode and mercury cathode to metals other than those dealt with in their first paper (ANALYST, 1905, xxx., 413). The apparatus used was the same as before, except that the weight of mercury used was increased to 60 or 70 grams. For *cadmium*, good results were obtained by electrolysing 5 c.c. of solution containing 0·9480 gram of cadmium as sulphate, and $\frac{1}{8}$ c.c. of concentrate sulphuric acid for fifteen minutes, with a current of 3·5 ampères and 10 to 7 volts. For *tin*, the best results were obtained with 10 c.c. of solution containing 0·8212 gram of tin as stannous sulphate, and 0·5 c.c. of sulphuric acid, using a current of 5 ampère and 5 to 4 volts for eight minutes. For *silver*, the best results were obtained with 5 c.c. of solution containing 0·2240 gram of silver as nitrate, and $\frac{1}{8}$ c.c. of nitric acid, using a current of 4·5 to 3 ampères at 6·5 to 6 volts for five minutes, and giving the anode the high speed of 1,200 revolutions per minute. No nitric acid was reduced and hence no ammonium was found in the amalgam. For *mercury*, 5 c.c. of solution containing 0·3575 gram of mercury as mercurous nitrate and 0·02 c.c. of nitric acid gave the best results with a current of 3 ampères and 7 to 5 volts for three minutes;

diluting the liquid to 25 c.c. increased the time necessary to eight minutes. For *bismuth*, the best conditions were: Volume, 12 c.c.; bismuth, as sulphate, 0·2273 gram; 0·5 c.c. of sulphuric or nitric acid; current, 3 or 4 ampères at 5 to 5·75 volts; time, fifteen minutes for sulphuric, and twelve minutes for nitric, acid solutions. The platinum wire used as anode should be very smooth, in order that the bismuth peroxide at first deposited on it may be readily dissolved.

Experiments to determine halogens in metallic haloid salts by using a silver-coated rotating anode proved unsuccessful, some of the deposit always becoming detached. It was found possible, however, to determine the metal in such salts, the free halogen being suppressed by adding 10 c.c. of toluene to the liquid. The most favourable conditions found are summarized in the following table:

Metal.	Amount taken (gram).	Volume of Liquid (c.c.).	Ampères.	Volts.	Time (minutes).	Maximum Error (gram).
As Chloride:						
Cobalt ...	0·1050	5	2 to 4	5	7	±0·0002
Gold ...	0·1200	5	2 to 3	10	5	−0·0002
Ferric ...	0·1030	5	2 to 4	9	12	−0 0005
Mercuric ...	0·2525	5	1 to 3	10 to 7·5	10	−0·0001
Stannous ...	0·0800	5	2 to 3	7 to 6	10	+0·0006
Ditto ...	0 1600	10	2 to 3	7 to 6	15	−0·0005
As Bromide:						
Cadmium ...	0·2212	5	2	5	10	+0·0003

The anode was not attacked in these experiments; xylene may be used instead of toluene, which is probably converted into a mixture of *o*- and *p*- chlortoluene.

It was also found possible to separate iron quantitatively in this way from uranium, aluminium, thorium, lanthanum, praseodymium, neodymium, cerium, and zirconium. The metals were used in the form of sulphates. The quantity of iron present was 0·12 to 0·18 gram, and of the sulphate of the other metal 0·1 to 0·5 gram. The volume of the liquid varied from 6 to 10 c.c., the quantity of free sulphuric acid from 0 to $\frac{1}{10}$ c.c., the current from 2 to 5 ampères at 9 to 5 volts, and the time from fifteen to twenty-five minutes. The maximum error in twenty-nine experiments was −0·0008 gram iron.

A. G. L.

Estimation of Vanadium. W. Heike. (*Chem. Zeit. Rep.*, 1905, xxix., 392.) —A comparison of four methods for purposes of technical iron works:

1. The ore is fused with sodium potassium carbonate, extracted with hot water, filtered, the filtrate acidified with H_2SO_4, the vanadic acid reduced to vanadic oxide with zinc, and the amount estimated by titration with standard permanganate. The vanadium titre = iron titre × 0·035.

2. The ore is dissolved, the iron extracted by shaking out with ether, the solution treated with H_2O_2, and the vanadium estimated by comparing the tint (yellow-red) with that of known vanadic solutions.

3. Ledebur dissolves the iron, etc., in diluted hydrochloric acid, separates the chromium and vanadium from iron and manganese by neutralizing with the barium carbonate, fuses the residue with sodium carbonate and nitre, and extracts the chromates and vanadates with hot water. The solution is then reduced with alcohol and hydrochloric acid, the chromium precipitated with ammonium phosphate and ammonia, the vanadium transformed by ammonium sulphate into the sulphide, and precipitated as sulphide with acetic acid. On heating vanadic acid is obtained.

4. Campagne extracts the iron with ether (as in 2), reduces the vanadic acid by evaporation with hydrochloric acid to V_2O_4, displaces the hydrochloric acid by sulphuric acid, warms to 60° to 70°, dilutes to 300 c.c., and titrates with potassium permanganate. The vanadium titre here = iron titre × 0·914.

The author obtained the following figures:

	I.	II.	III.	IV.
Iron ore	0·07	—	—	0·11
Fresh slag	1·40	—	—	1·46
Gray crude iron with 0·14 Cr.	0·15	—	0·21	0·21
Steel with 0·02 Cr.	—	0·24	0·27	0·54
Steel	—	0·42	0·54	0·54

He concludes by stating that Campagne's method is very suitable for estimation of ferrovanadium, especially where chromium is not present in large quantity, otherwise Ledebur's method is to be preferred. E. K. H.

Moisture and Water of Constitution in Bauxite. H. Lienau. (*Chem. Zeit.*, 1905, xxix., 1280.)—The view has often been expressed that the moisture in bauxites must be estimated by drying in the desiccator and not in the oven, as the water of hydration begins to come off even at 100° C.

The author performed experiments to test this view. The powdered bauxite was wetted with 14 to 15 per cent. of distilled water, and five samples of 10 to 15 grams were placed in stoppered weighing bottles and dried for ten hours at 100°, allowed to stand overnight in a desiccator, and weighed. They were then dried for ten hours at 110°, allowed to stand as before, and again weighed. This was repeated for temperatures of 150° and 200°. It might then be supposed that a characteristic gap would occur between the loss of the moisture and that of the water of constitution. As a matter of fact, for most kinds of bauxite no such gap could be distinguished, but the loss of water was continuous with rise of temperature, until the whole of the water of hydration was evolved, and a constant weight obtained at bright red heat.

The term "bauxite" includes materials of very different constitution, but the French varieties at least (on which the experiments were performed) can be divided into two great classes: (1) The diaspore group, containing alumina monohydrate, and (2) the true bauxite group, containing alumina dihydrate. The author gives figures for both classes, for which reference must be made to the original. The conclusions arrived at may be summarized as follows:

For the monohydrate group, which forms about 90 per cent. of commercial bauxites, the weight alters evenly up to 200° C., but so slowly that the loss in weight between 100° and 200° is only 0·09 per cent. It is not possible to say at what point the combined water begins to be given off, but it is probably fair to assume that there is no more adherent moisture at 110°, as the loss between 100° and 110° = 0·02, whilst loss between 110° and 150° = 0·03 per cent. This is further made more likely by the behaviour of the dihydrate group.

One specimen of this class from Maussane, near Arles, showed a distinct gap between the two kinds of water, the weight remaining constant between 110° and 150°. The author considers this characteristic, and thinks that in these bauxites the water of hydration only begins to come off after all the adherent moisture has been driven off.

The author concludes that in all bauxites the total loss in weight up to 110° C. may be taken as adherent moisture. E. K. H.

Volumetric Estimation of Sulphates in Presence of Thiosulphates, etc. O. Huber. (*Chem. Zeit.*, 1905, xxix., 1227.)—The author investigated the process originally brought forward by Müller and Dürkes for the volumetric estimation of sulphates with benzidin hydrochloride, and extended it to the estimation of sulphates when thiosulphates, sulphites, and sulphides were also present. The only modification made was the substitution of caustic potash for caustic soda. The author gives figures which show an error of about 2 per cent. in value for total sulphate, and states that this process is inaccurate for the following reasons:—The use of so many titrations, the errors of which may all lie in the same direction, but chiefly, in the author's opinion, through the defects of the benzidin process itself. He concludes that the chief factor in rendering the process inexact is the great solubility of the benzidin sulphate, whilst the absorption phenomena noted by Müller and Dürkes are less important.

For further details and the figures given reference must be made to the original.

E. K. H.

Estimation of Sulphuric Acid by Means of Barium Chloride in the Presence of Disturbing Substances. G. Lunge and R. Stierlin. (*Zeit. angew. Chem.*, xviii., 1921.)—The authors come to the conclusion that the precipitation of barium chloride as sulphate is by no means complete in the presence of many salts. Thus, if sodium, potassium, or ammonium salts are present, part of the barium sulphate is held in solution. Zinc and copper compounds may, however, be present without disturbing the reaction in any way. This undesirable influence is negligible, however, if the precautions suggested by Lunge (*Zeit. anal. Chem.*, 1881, vol. xix., p. 149) are adhered to. H. D. L.

The Determination of Nitrous and Nitric Acids. J. Meisenheimer and F. Heim. (*Berichte*, 1905, xxxviii., 3834-3837.)—This method, which is claimed to give rapid and accurate results in the simultaneous determination of nitrous and nitric acids, is based upon the following reactions:

$$HNO_2 + HI = NO + I + H_2O$$
$$HNO_3 + 3FeCl_2 + 3HCl = NO + 3FeCl_3 + 2H_2O.$$

The slightly alkaline solution of the substance (0·1 to 0·2 gram of nitrite) is placed in a round-bottomed 50 c.c. flask closed by an indiarubber stopper with three openings. One of these receives a thistle funnel with a stop-cock; the second holds a delivery-tube, the outside portion of which dips into a trough containing a 12 per cent. solution of sodium hydroxide; whilst the third receives a tube for the introduction of a current of carbon dioxide. The air is first swept out of the flask by means of this gas for about ten minutes, after which a eudiometer filled with sodium hydroxide solution is fixed above the delivery-tube, and then by means of the funnel, the tube of which has previously been filled with water up to the stop-cock, there are introduced 10 to 15 c.c. of a 5 per cent. solution of potassium iodide, followed by the same quantity of dilute hydrochloric acid, added little by little. When the evolution of gas slackens, the contents of the flask are heated to incipient boiling, and the last portions of nitric oxide expelled by means of a current of carbon dioxide. The amount of gas contained in the eudiometer is then measured in the usual manner.

For the determination of nitrates in the residue, from 10 to 20 c.c. of a concentrated solution of ferrous chloride, acidified with hydrochloric acid, are introduced into the flask, and the gas collected as described in Spiegel's modification of the method of Schulze and Tiemann (*Berichte*, 1890, xxiii., 1361). C. A. M.

The Determination of Nitrous Acid. F. Raschig. (*Berichte*, 1905, xxxviii., 3911-3914.)—The method described by Meisenheimer and Heim (see preceding abstract) has been applied by the author to the determination of nitrous acid in solutions containing also hydroxylamine, but instead of measuring the nitric oxide in a eudiometer he titrates the separated iodine, a determination being made in five to ten minutes. The solution (about 100 c.c.) is placed in a 200 c.c. conical flask, into which are then introduced 5 to 10 c.c. of a 10 per cent. solution of potassium iodide. A current of carbon dioxide is now admitted through a tube passing nearly to the bottom of the flask, and after two or three minutes about 1 c.c. of $\frac{N}{10}$ sulphuric acid is poured down the side of this tube, and the mixture allowed to stand for two minutes, and then titrated with the standard thiosulphate solution, which is also made to run down the side of the tube. The bubbles of gas evolved cause the liquids to mix, and thus the flask need not be shaken, which would cause oxidation of the escaping nitric oxide. In the author's opinion nitrosyl iodide (NOI) is first formed on adding the acid, and this is then decomposed, so that it is necessary to wait for a time before titrating the solution; otherwise the results will be too high. The author prefers the sulphanilic acid and permanganate methods where possible, but they cannot be employed in the presence of hydroxylamine. As regards the permanganate method, he uses an excess of at least 20 per cent. of $\frac{N}{10}$ permanganate solution, into which the solution under examination is poured. The mixture is acidified, if required, and allowed to stand for two minutes, when the whole of the nitrous acid will have been oxidized to nitric acid. The liquid is then treated with 5 c.c. of a 10 per cent. solution of potassium iodide, which dissolves any manganese peroxide, and the liberated iodine titrated with standard thiosulphate solution. C. A. M.

The Reactions of the three Phosphoric Acids. C. Arnold and G. Werner. (*Chem. Zeit.*, 1905, xxix., 1326, 1327.)—The authors have tested all the reactions described in chemical text-books as means of distinguishing between the three phosphoric acids, and have found that in nearly every instance they are unreliable. They have therefore made a new series of tests with the alkali salts of the acids, and the principal results obtained are summarized in the following table:

REAGENT.	ORTHOPHOSPHORIC ACID.	PYROPHOSPHORIC ACID.	METAPHOSPHORIC ACID.
Barium chloride	White ppt. Insol. in excess of alkali phosphate. Sol. in acetic acid. Sol. in NH_4Cl.	White ppt. Insol. in excess of alkali pyrophosphate. Insol. in acetic acid. Insol. in NH_4Cl.	ppt. of alkali metaphosphate. Insol. in acetic acid. Insol. in NH_4Cl.
Calcium chloride	White ppt. Insol. in excess of phosphate. Sol. in acetic acid. Sol. in much NH_4Cl.	White ppt. Scarcely sol. in excess of pyrophosphate. Sol. in acetic acid. Insol. in NH_4Cl.	White ppt. Sol. in excess of metaphosphate. Insol. in acetic acid. Insol. in NH_4Cl.
Magnesium sulphate	White flocculent ppt. Insol. in excess of phosphate and in excess of $MgSO_4$. Sol. in acetic acid.	White ppt. Sol. in excess of pyrophosphate. Sol. in acetic acid. Insol. in NH_4Cl.	White ppt. Sol. in excess of metaphosphate and in excess of $MgSO_4$. Insol. in acetic acid.
Magnesia mixture	White crystalline ppt. Insol. in excess of phosphate and of reagent. Readily sol. in acetic acid.	White ppt. Sol. in excess of pyrophosphate and of reagent. Sol. in acetic acid. In very dilute solutions ppt. only formed after heating.	White ppt. Sol. in excess of metaphosphate. Insol. in excess of reagent and in acetic acid.
Aluminium sulphate	White ppt. Sol. in excess of phosphate and in acetic acid.	White ppt. Sol. in excess of pyrophosphate. Insol. in acetic acid.	White ppt. Sol. in excess of metaphosphate. Insol. in acetic acid.
Chromium sulphate	Greenish ppt. Insol. in excess of phosphate. Sol. in acetic acid.	Dirty-white ppt. Insol. in excess of pyrophosphate and in acetic acid.	Dirty white ppt. Sol. in excess of metaphosphate. Insol. in acetic acid.
Ferrous sulphate	Green ppt. Insol. in excess of phosphate.	White ppt. Sol. in excess of pyrophosphate.	—
Ferric chloride	Yellow ppt. Insol. in excess of phosphate. Sol. in HCl.	White ppt. Hardly sol. in excess of pyrophosphate. Sol. in HCl.	White ppt. Sol. in excess of metaphosphate and in much HCl.
Manganese sulphate	White ppt. Insol. in excess of phosphate. Presence of much pyrophosphate prevents ppt.	White ppt. Hardly sol. in excess of pyrophosphate.	White ppt. Readily sol. in excess of metaphosphate.
Zinc sulphate	White ppt. Insol. in excess of phosphate. Sol. in NH_3 and in acetic acid. Metaphosphate does not prevent ppt.	White ppt. Sol. in excess of pyrophosphate. Sol. in NH_3. Insol. in acetic acid. Metaphosphate does not prevent ppt.	
Cobalt nitrate	Blue ppt. Insol. in excess of phosphate. Sol. in acetic acid.	Rose ppt. Sol. in excess of pyrophosphate. Insol. in acetic acid.	Rose ppt. Sol. in excess of metaphosphate. Insol. in acetic acid.
Nickel sulphate ...	Greenish-white ppt. Insol. in excess of phosphate. Sol. in acetic acid.	Greenish-white ppt. Sol. in excess of pyrophosphate. Sol. in acetic acid.	—

REAGENT.	ORTHOPHOSPHORIC ACID.	PYROPHOSPHORIC ACID.	METAPHOSPHORIC ACID.
Uranium nitrate $(UO_2)(NO_3)_2$	Yellow ppt. Insol. in excess of phosphate.	White ppt. Sol. in excess of pyrophosphate.	White ppt. Sol. in excess of metaphosphate.
Silver nitrate	Yellow ppt. Insol. in excess of phosphate. Sol. in HNO_3 and NH_3.	White ppt. Insol. in excess of pyrophosphate. Sol. in HNO_3 and NH_3.	White ppt. Sol. in excess of metaphosphate. Sol. in HNO_3 and NH_3.
Lead acetate	White ppt. Insol. in excess of phosphate. Sol. in KOH and HNO_3. Sol. in hot dilute HCl. Sol. in much tartaric acid.	White ppt. Sol. in excess of pyrophosphate and in KOH, HNO_3, hot dilute HCl, and much tartaric acid.	White ppt. Sol. in excess of metaphosphate, in KOH, HNO_3, and hot dilute HCl. Insol. in tartaric acid.
Mercurous nitrate ...	Yellowish-white ppt. Insol. in excess of phosphate. Sol. in HNO_3.	White ppt. Sol. in excess of pyrophosphate. Sol. in very much HNO_3.	White ppt. Sol. in excess of metaphosphate. Hardly sol. in HNO_3.
Mercuric chloride ...	Immediate yellow ppt. In concentrated solution yellow turbidity, and deep violet-red ppt. after some hours.	In concentrated solution red fluorescent turbidity after a few minutes. Then reddish-brown ppt.	—
Alkaline bismuth solution	—	—	White ppt. (also in presence of ortho- and pyrophosphate). Sol. in excess of metaphosphate.
Copper sulphate ...	Greenish-blue ppt. Insol. in excess of phosphate. Sol. in NH_3 and acetic acid.	Bluish-white ppt. Sol. in excess of pyrophosphate and NH_3. Insol. in acetic acid.	—
Cadmium sulphate...	White ppt. Insol. in excess of phosphate. Sol. in NH_3 and in acetic acid.	White ppt. Insol. in excess of pyrophosphate. Sol. in NH_3. Insol. in acetic acid.	White ppt. Sol. in excess of metaphosphate. Sol. in NH_3. Insol. in acetic acid.

For the detection of metaphosphoric acid in the presence of the other two acids, the authors consider the reactions with alkaline bismuth solution (*vide supra*) and with cobaltamine solution particularly suitable. The latter reagent is prepared by mixing equal parts by volume of solutions of a cobaltous salt and an ammonium salt, adding a few drops of ammonium hydroxide, and shaking the solution until it becomes perceptibly brown. It gives a brownish-yellow precipitate with a metaphosphate even when only present in traces in ortho- and pyrophosphates. Pyrophosphoric acid in the presence of the other acids is best identified by its behaviour with copper sulphate and zinc sulphate in acetic acid solution.

C. A. M.

Estimation of Phosphoric Acid in Thomas Meal, Bone Meal, etc. O. Böttcher. (*Chem. Zeit.*, 1905, xxix., 1293.)—1. *Estimation of Citric Acid Soluble Phosphate in Thomas Meal.*—The author maintains, against various critics, that, as agreed upon by the Union of Agricultural Research Stations (in Germany), the phosphate can be directly precipitated without error with magnesia mixture containing citrate, without previous separation of silica, by evaporation with hydrochloric acid, even in cases where a high percentage of silica is shown by a preliminary test. The process gives accurate results so long as the essential condition is observed that all the operations are carried through successively without interruption. If the citric acid extracts, or the precipitations with citrate-containing magnesia mixture, are allowed to stand for hours, considerable quantities of silica may come down with the phosphate precipitate and cause a too high result. The separation of silica is always indicated by the bad filtering property of the solution, and this serves as a valuable check on the purity of the precipitate. The author has carried out analyses on over 800 samples of Thomas meal in the last year, and in no case encountered this complication.

The author gives figures which show very exact agreement between the results obtained by this direct estimation and those obtained by first separating the silica.

2. *Estimation of Total Phosphate in Thomas Meal, Bone Meal, etc.*—The author again defends the process prescribed by the Union of Agricultural Research Stations: (1) Against the criticism of V. Schenke, who, finding 0·3 to 0·4 too little phosphoric acid by this method, as against the molybdate method, recommended approximately neutralizing the strongly acid solution before precipitation; and (2) against those analysts who consider that extraction with aqua regia gives less reliable results than extraction with sulphuric acid, partly because the compensation in the aqua regia solution varies with the kind and amount of bases present, and partly because all the organic acids are not decomposed by the aqua regia, so that the calcium salts of these acids accompany the precipitate, being insoluble in ammonium citrate solution, the author gives figures of comparative analyses which show that the modifications suggested by these criticisms make no appreciable difference, and that the Union process is quite satisfactory. E. K. H.

The Determination of Tellurous and Telluric Acids. A. Berg. (*Bull. Soc. Chim.*, 1905, xxxiii., 1310-1312.)—The author's method depends upon the facts that these acids are readily converted into tellurium chloride by the action of gaseous hydrochloric acid, and that the chloride formed can easily be sublimed. The apparatus required is a combustion tube of hard glass, the front part of which is bent at a right angle and drawn out to a fine opening, which is directed downwards and connected with two small U-tubes containing water. The tube is placed in a combustion furnace and its other end connected with an apparatus giving a current of hydro chloric acid gas. The substance to be analysed is placed in a porcelain boat which is introduced into the tube. The air is swept out by means of a moderate current of the gas, and the tube rapidly heated to a temperature below red heat. The tellurium chloride which sublimes on the tube is driven forward by the application of a Bunsen burner. If the process is properly carried out no white fumes should be seen in the absorption tubes, and the liquid in the second tube should remain colourless. When the reaction is complete, the yellow solution of tellurium chloride is transferred to a weighed porcelain crucible, and after the addition of 5 c.c. of nitric acid evaporated slowly on a sand bath. The presence of this nitric acid prevents the volatilization of the tellurium chloride. When the evaporation has proceeded to dryness, the residue must be cautiously heated so as to decompose all the tellurium nitrate without fusing the tellurous acid, part of which would then escape with the nitrous vapours. With a little care this can readily be done. The residue is then weighed as tellurous anhydride. When metallic tellurites or tellurates are to be analysed, the residue of chloride left in the boat can be weighed directly. In a test experiment 58·66 per cent. of tellurous anhydride was found as compared with the theoretical 58·26 per cent. A barium tellurite containing, according to theory, 60·94 per cent. of tellurous anhydride was found to contain 60·28 per cent. The method is obviously only applicable in the absence of compounds of substances, such as mercury or chromic acid, which yield readily volatile compounds under these conditions.

C. A. M.

The Volumetric Estimation of Cyanates. A. C. Cumming and Orme Masson. (*Chem. News*, 1906, vol. 93, pp. 5 and 17.)—A known volume of the cyanate solution is first of all titrated in the cold with standard acid, using congo-red or methyl orange as indicator; the first end-point obtained should be taken. This gives the quantity of carbonate present, carbonate being always contained in cyanate solutions. An excess of the acid is then added, and the liquid boiled for a few minutes, when the cyanate is hydrolysed according to the equation:

$$KCNO + 2HCl + H_2O = KCl + NH_4Cl + CO_2.$$

The liquid is then cooled and the excess of acid titrated back with standard alkali, the quantity consumed being calculated to cyanate. As a further check the ammonia formed may be determined by boiling the liquid for some time with an excess of standard alkali and titrating back. The method appears to give good results. If cyanide is present it is determined by means of standard silver nitrate, and allowed for in calculating the results. Ferrocyanide in small quantities does not interfere. The solutions used should be rather dilute, as otherwise the acid might act on the cyanate in the cold, and render the first end-point indefinite.

A. G. L.

Determination of Cyanides in Crude Coal-Gas. (*Chem. Trade Jour.*, 1906, xxxviii., 72, 73.)—In the proposed method, the cyanides are absorbed by freshly precipitated ferrous hydroxide suspended in sodium hydroxide solution. This is prepared by dissolving 4 grams of crystallized ferrous sulphate in 500 c.c. of water at a temperature of 80° C., adding 4 grams of sodium hydroxide dissolved in water also at 80° C., and boiling the mixture. The precipitate is allowed to settle, washed by decantation, and then mixed with 250 c.c., of 4 per cent. sodium hydroxide solution. Water is finally added to make the volume up to about 800 c.c., and the mixture transformed to an absorption apparatus, which consists of a tall glass jar closed at the top with a caoutchouc stopper perforated by two holes. The central one of these carries a length of half-inch solid-drawn nickel tubing reaching to the bottom of the cylinder, and upon which are brazed twelve perforated nickel cones. The liquid should fill the cylinder up to the base of the topmost cone. Ten cubic feet of gas are usually sufficient for one test, and this volume is passed through the apparatus at the rate of 5 cubic feet per hour. At the end of the operation the contents of the cylinder are washed out into a litre flask and diluted to the mark. A known volume of this solution is then boiled to expel ammonia, then treated with sodium plumbate to remove sulphide (in this operation free sodium hydroxide must be present), made up to the correct volume, and the amount of ferrocyanide formed determined in it. W. P. S.

Purification of Hydrochloric Acid from Arsenic. (German Patent, *Apoth. Zeit.*, xx., 932; through *Pharm. Jour.*, 1905, lxxv., 910.)—Vanadous salts rapidly reduce arsenious chloride to metallic arsenic, which separates from the solution in the form of minute particles, or as a flocculent precipitate which can easily be filtered off. On a large scale the hydrochloric acid gas is passed through earthenware vessels containing a concentrated solution of vanadous chloride, which retains the arsenic, and is simultaneously oxidized to vanadic chloride. The latter can be reconverted into vanadous chloride. W. P. S.

A New Method of Determining the Strength of Iodine Solutions. Sigmund Metzl. (*Zeits. anorg. Chem.*, 1906, xlii., 156.)—The author proposes to standardize iodine solutions used for titrating antimony compounds by means of tartar emetic, either as the crystallized salt, $KSbO(C_4H_4O_6) + H_2O$, or in the anhydrous condition. The tartar emetic is dissolved in hot water containing tartaric acid, a trace of solid phenolphthalein and an excess of sodium carbonate are added, after which CO_2 is led through the liquid until the red colour is destroyed. The titration is then made as usual with the iodine solution to be standardized. The results obtained are in good agreement with those obtained by standardizing a thiosulphate solution with potassium bichromate in the usual way, and titrating the iodine against the thiosulphate. The author also recommends the use of potassium hydrogen iodate, which reacts with potassium iodide as follows in acid solution:

$$KH(IO_3)_2 + 10KI + 11HCl = 6I_2 + KCl + 6H_2O$$

and in neutral solution according to the equation:

$$6KH(IO_3)_2 + 5KI = 3I_2 + 11KIO_3 + 3H_2O,$$

whilst its purity can be easily determined by titration with alkali. A. G. L.

Tests and Reactions of Hydrogen Peroxide. C. Schmatolla. (*Pharm. Zeit.*, 1905, l., 641; through *Pharm. Jour.*, 1905, lxxv., 910.)—Ten c.c. of hydrogen peroxide should not require more than 0·25 c.c. of $\frac{N}{10}$ alkali for neutralization, using Congo red as an indicator. The presence of hydrochloric acid is very objectionable in samples intended for medicinal use, and hydrogen peroxide containing more than 0·01 per cent. of chlorine should be rejected. To determine this impurity, 10 c.c. of the sample are treated with 25 drops of dilute sulphuric acid, 0·5 gram of ferrous sulphate, and 5 c.c. of $\frac{N}{10}$ silver nitrate solution. The silver is then titrated back with $\frac{N}{10}$ thiocyanate solution.

The following test will detect hydrogen peroxide in a dilution of 1 : 1,000,000: 200 c.c. of the solution to be tested are acidified with a few drops of dilute sulphuric acid, and about the same quantity of a 1 per cent. cobalt nitrate solution is added. On then adding potassium hydroxide solution drop by drop, a sharp brown colour-reaction is produced if hydrogen peroxide be present. W. P. S.

A Substitute for Hydrogen Sulphide. J. Ducommun. (*Chem. Zeit. Rep.*, 1905, xxix., 379.)—The acidified solution of the metal or metallic salt is treated with 2 to 5 c.c. of formalin solution, and then with a moderately concentrated solution of pure sodium sulphide. If a metal of the arsenic-tin group be present, the corresponding sulphide is at once precipitated. On adding alkali to the filtrate, the precipitate of the iron group is obtained. The addition of the formalin prevents any separation of sulphur. E. K. H.

Determination of Oil in Paraffin-Scale. L. Neustadtl. (*Chem. Zeit.*, 1906, xxx., 38.)—In the method proposed, advantage is taken of the insolubility of paraffin in acetone at a temperature of - 15° C., whilst the oil present is completely soluble. One gram of the finely-divided paraffin-scale is mixed with 10 c.c. of acetone, and allowed to stand for two hours at the ordinary temperature, with occasional shaking. The mixture is then cooled to - 15° C., and the solid paraffin collected on a small plug of cotton-wool contained in a filter-tube. The latter is surrounded by a funnel, and the space between the two filled with a freezing mixture. After washing the filter and its contents with acetone cooled to - 15° C., the filtrate and washings are evaporated, the residue is dried at 100° C. and weighed. The results obtained are, as was to be expected, somewhat higher than those yielded by the older methods in which the oil was pressed out. W. P. S.

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REVIEW.

SELECT METHODS IN FOOD ANALYSIS. By DRS. LEFFMAN and BEAM. (London: Rebman. Price 11s. net.)

Since the publication in 1901 of this very useful handbook considerable additions have been made to our knowledge of food chemistry, and many new processes have been devised. This progress, to which American chemists have contributed a very full share, has necessitated a revision of the work and the insertion of much new matter. Among the more important additions are descriptions of useful distillation and extraction processes; new methods for the detection of natural colours used as substitutes for fruit colours; improvements in the detection and estimation of formaldehyde, boric acid, and other preservatives; and some new information in connection with the analytical chemistry of butter, oleo-margarine, and other fats. The arrangement of the book leaves nothing to be desired, and although the descriptions of analytical processes are invariably brief, in very few cases has compression been secured at the expense of clearness. The estimation of minute traces of arsenic in food products is a matter with which American chemists are perhaps not quite so intimately acquainted as their English colleagues, and the section devoted to this subject is capable of being improved.

On p. 62 the operator is told to use 3 grams of zinc in the Marsh-Berzelius flask, and to allow the action to proceed for one hour after the addition of the material to be tested. It is not quite clear how a sufficient supply of gas is to be obtained by the employment of so small an amount of metal in a test which, according to the authors' instructions, is to last for rather more than one and a half hours. Nothing, moreover, is said to warn the analyst against the use of "insensitive" zinc. On p. 91 the size of the pepper-starch granules is curiously said to vary from 0 to 5·5 microns. Mention is made of the use of cocoanut oil as a direct or indirect butter adulterant, but it is to be regretted that the authors do not give more detailed and definite information as to the methods to be employed for its detection. The illustrations are as a rule good, the plate showing the microscopical appearance of certain of the starches being one of the least satisfactory. To the English eye such words as *sirup* and *analogs* have a strange appearance, but the spelling may perhaps be defended on the ground of simplicity, and at least does no violence to the principles of etymological science. The same cannot, however, be said for the word *levulose.*

The work is described in the authors' preface as being intended "for the practical worker in the detection of food adulteration," and all such will welcome it as one of the best contributions to this important subject which has yet appeared.

A. C. C.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

ANNUAL MEETING.

THE twenty-eighth Annual General Meeting of the Institute of Chemistry of Great Britain and Ireland was held at 30, Bloomsbury Square, on Thursday, March 1, Mr. David Howard, the retiring President, in the chair.

The accounts for 1905 having been submitted by the HON. TREASURER (Mr. A. Gordon Salamon), and duly received, Dr. JOHN A. VOELCKER moved the adoption of the Annual Report. He congratulated the Council on their work during the past year, and he specially alluded to the steps they had taken to protect the interests of professional chemists. Mr. ARTHUR E. EKINS seconded, and the Report was formally received and adopted.

Scrutineers having been appointed to count the votes sent in for the election of the officers and members of Council, and ballot having been taken for the election of censors, the meeting proceeded to select the honorary auditors, and Messrs. Robert E. Alison, W. T. Burgess, and P. A. E. Richards were appointed.

The PRESIDENT then delivered his address. After referring to the progress made by the Institute during his term of office as President, he dealt more particularly with the history of the third and last year.

While the roll of Fellows and Associates has steadily increased, he noted with regret the death of some of the original Fellows, and he mentioned especially John Lloyd Bullock, largely to whose incentive was due the foundation of the Royal College of Chemistry and the invitation to Hofmann to come to London. How much that meant to not a few, including several past Presidents of the Institute, it would take long to tell.

The Institute was examining over one hundred candidates yearly, and arrangements would be made from time to time for examinations in the colonies.

Mr. **Howard** alluded to the improvement in the financial position of the Institute, and to the steady growth of the library. He paid a high tribute to the services of Professor Adrian J. Brown, who had held the appointment of Examiner to the Institute in Biological Chemistry since 1901, and stated that, as his term of office had expired, the Council had selected Dr. Arthur Harden, of the Lister Institute, as his successor.

He also referred to the new examinations in technical chemistry, the first of which will be held in October next. In this connection he expressed the hope that the Institute would in future have for its Presidents industrial chemists, as well as those eminent for educational and strictly professional work. The chief function of the Institute was to register competent consulting, analytical and technical chemists, and the Institute might safely claim to represent the profession. The Council regarded it as their duty to advance the interests of the profession, and, as far as they were able, to maintain it on a sound and satisfactory basis. They had lately been obliged to make representations to authorities whose actions appeared to be detrimental to the profession.

With reference to the gratuitous performance of analyses at agricultural colleges, he mentioned that the Board of Agriculture had endeavoured to show that the performance of cheap milk tests had been arranged for educational purposes. At the same time the Board had stated that these tests were not seriously to be compared with the analyses made by public analysts and district analysts. The Council had to complain of more than the milk tests: the colleges were undertaking all kinds of analyses at nominal fees—of soils, for instance, at half a crown. This was undoubtedly injurious to the profession; but the Board, in its journal, stated that "the demand on the part of landowners for expert advice from the lecturers had been considerably in excess of what might reasonably have been anticipated." In this matter the Council had not received a satisfactory response.

He referred to the great advances in chemistry that had been due to the work of private practitioners, giving his opinion that any action which tends to interfere with the individual practitioners would be fatal to progress.

It was with reluctance that the Council had to take up an attitude which might seem in any way antagonistic to the National Physical Laboratory, but they had been obliged to direct the attention of the Executive Committee to the fact that their test pamphlet indicated that they might undertake work which they were forbidden to undertake under the Treasury Report on which the laboratory was founded. The Executive Committee has recently given their assurance of their desire to avoid any cause for complaint. He was sure that the Council would be glad to see the laboratory placed on such a sound footing financially that its authorities would have no temptation to extend the work of the laboratory beyond its proper sphere.

Mr. **Howard** thought the profession had reached a somewhat critical stage in its history. With greater facilities for training and, consequently, a larger supply of chemists, it was evident that only the most efficient could hope to be successful, He believed the demand for chemists was increasing, but authorities and manufacturers were learning that they must have efficiency.

After dealing briefly with the present position of the industrial chemist and the official professional chemist, he referred to the professors and teachers of chemistry. He objected to the practice, which had grown of late, of blaming the Universities for the loss of certain industries. He maintained that, while chemists were improving so greatly in efficiency, it was absurd to blame the professors. Every decade does not bring a Hofmann, but there were many teachers at the present day under whom the bulk of Fellows and Associates were proud to say they had been trained.

The Institute afforded a great benefit to the public by aiding its discrimination in the selection of competent chemists of acknowledged ability and professional integrity; they could rely on each Fellow and Associate to further the reputation of the Institute by his character and conduct, the soundness of his work, and by the cultivation of professional feeling.

Mr. HOWARD then referred to the new President, Professor Percy F. Frankland, who had long been associated with the Institute, and whose father, Sir Edward Frankland, was the founder and first President. He concluded by saying how much he had appreciated his position as President of the Institute, and he spoke in warm terms of the co-operation of the Councils with whom he had worked

A vote of thanks for the address was moved by Mr. THOMAS TYRER, seconded by Professor J. MILLAR THOMSON, supported by Mr. R. J. FRISWELL, and carried unanimously.

On the report of the scrutineers the result of the balloting for the selection of censors was submitted, and Mr. David Howard; Sir William Ramsay, K.C.B., F.R.S.; Thomas Stevenson, M.D.; and Professor J. Millar Thomson, F.R.S., were declared elected.

The officers and members of Council for the ensuing year were then declared elected, as follows:

President: Percy Faraday Frankland, LL.D., Ph.D., F.R.S. *Vice-Presidents:* Edward John Bevan; Edward Divers, M.D., D.Sc., F.R.S.; David Howard; Edmund Albert Letts, D.Sc.; Edmund James Mills, D.Sc., F.R.S.; Sir William Ramsay, K.C.B., LL.D., F.R.S. *Hon. Treasurer:* Alfred Gordon Salamon, A.R.S.M. *Members of Council:* Adrian John Brown, M.Sc.; Lieut.-Colonel Charles Edward Cassal; Arthur Crozier Claudet, A.R.S.M.; John Norman Collie, Ph.D., F.R.S.; James Kear Colwell; Cecil Howard Cribb, B.Sc.; Henry John Horstman Fenton, M.A., F.R.S.; Martin Onslow Forster, D.Sc., F.R.S.; Richard John Friswell; William Gowland, A.R.S.M.; Arthur George Green; Henry George Greenish; Oscar Guttmann; James Hendrick, B.Sc.; Egbert Grant Hooper; Herbert Jackson; Arthur Robert Ling; Henry de Mosenthal; Henry Droop Richmond; Alfred Smetham; Arthur Smithells, B.Sc., F.R.S.; John Edward Stead, F.R.S.; David Alexander Sutherland; Francis Napier Sutton; Edward William Voelcker, A.R.S.M.; William Palmer Wynne, D.Sc., F.R.S.; Sydney Young, D.Sc., F.R.S.

On the motion of Lieut.-Colonel CHARLES E. CASSAL, seconded by Mr. P. GERALD SANFORD, a vote of thanks was accorded to the retiring officers and members of Council. The PRESIDENT having replied, the meeting terminated.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE PURIFICATION OF ZINC AND HYDROCHLORIC ACID FROM ARSENIC.

By L. T. Thorne, Ph.D., and E. H. Jeffers.

(*Read at the Meeting, February* 7, 1906.)

1. *Zinc.*—Hehner first suggested (Analyst, 1902, 261) the treatment of zinc with sodium for the purpose of removing traces of arsenic, but the method was somewhat uncertain, owing to the exact conditions necessary not being ascertained. Later we described before this Society conditions of this treatment under which we obtained reliable results; but as the manipulation we then used was complicated, we delayed printing the details, in order, if possible, to simplify it. After long trials and use we now lay before the Society the simplest conditions which we find reliable.

Commercially pure zinc is melted in a crucible in a gas furnace, and when at or only just above its melting-point sodium is mixed with it in the proportion of about 1 gram to each pound of zinc. This is best effected by adding the sodium in pieces of 1 to 2 grams weight which have not been dried from the adhering petroleum in which the sodium is stored. If the zinc be at the right temperature (*i.e.*, not too hot) the sodium is dissolved without either it or the petroleum vapour becoming ignited. After all the sodium has been added, it is preferable to add a small piece of zinc to insure the temperature being reduced to about the melting-point. The crucible is heated for a few minutes till the zinc is completely fluid, and the zinc is then poured into a second previously heated crucible, and back again once or twice to insure thorough melting. If the temperature is not allowed to get too high practically no oxidation takes place during this transference. The crucible, with the lid on, is then put back into the furnace and heated to a dull red heat, when the furnace and crucible lids are both removed, and the heating is continued for at least half an hour (preferably for an hour), care being taken that the temperature should not rise much. A scum (or alloy) gradually rises to the top and forms a hard, dark gray crust on the surface about $\frac{1}{8}$ inch thick. This crust contains practically all the arsenic and the greater part of the sodium in combination with zinc. The crucible is allowed to cool slightly, the crust is pierced at one side, and the molten mass is poured into a second previously heated crucible and skimmed if necessary. This crucible is then heated to bright redness, any slight scum removed, then allowed to cool, and the zinc granulated just before the solidifying-point is reached.

The essential points for success appear to be to get as thorough mixing as possible at a low temperature, and to avoid material rise in temperature whilst the scum or alloy is forming and rising. The arsenic sodium and zinc compound seems to be most stable at somewhere near the melting-point of zinc, and to be partially broken up at high temperatures. The temperature, before the scum is removed, should not be hot enough for the zinc or scum to take fire when the crucible lid is removed.

In this way zinc absolutely free from arsenic is obtained, which gives in the Marsh-Berzelius apparatus a perfect blank with the solution of 15 to 20 grams of zinc, and is yet so sensitive as to give a definite and reliable result with 0·000002 grams of arsenious oxide, and the sensitiveness is always the same. The yield is about 95 per cent. of the zinc taken.

2. *Hydrochloric Acid.*—In 1902 we described (*Proc. Chem. Soc.*, 1902, 118) a method for the freeing of hydrochloric acid from arsenic by a modification of the Reinsch test. In use we found the presence of a little stannous chloride (*i.e.*, of a strong reducing agent) greatly facilitated the deposition of the arsenic on the copper gauze, lessened the amount of copper dissolved, and shortened the process, but it still remained somewhat tedious. Noticing the great effect of the tin, we then tried the employment of a copper-tin couple with complete success.

Redistilled hydrochloric acid is diluted to rather under a specific gravity of 1,100 (water = 1,000), and is then poured on to a copper-tin couple, prepared by reducing with zinc a mixture of copper and tin chlorides in hydrochloric acid and washing the precipitated spongy couple. The metals are used in about the proportion of 1 part by weight of tin to 4 to 8 parts by weight of copper. A convenient mode of preparing the couple is as follows: Cuprous chloride is dissolved in excess of hydrochloric acid, and a small quantity of granulated tin added. The tin rapidly dissolves, at the same time reducing some copper in a spongy form. Zinc dust, or the smaller portions of granulated zinc, is then added, and this reduces the remainder of the copper together with the tin in the form of a dark gray spongy couple, which is washed by decantation. Ordinarily pure cuprous chloride and tin may be used, as traces of arsenic which may be present are eliminated, and do not affect the final result. The couple may also be readily obtained by reducing with zinc the residual liquor in the distilling flask, which consists of the mixed chlorides. The couple should be gray in colour; if it is nearly black the copper is in too great excess. About 2 or 3 grammes of the couple are used to each litre of the diluted acid. The acid containing the couple is gradually heated, a strong reaction taking place at about 70° C., and kept on the boil for about a quarter to half an hour. During this time most of the tin and a part of the copper is dissolved, and practically the whole of the arsenic is evolved. The acid may then be distilled direct, or be cooled and kept under a layer of petroleum (to prevent absorption of oxygen); but in the latter case it must be distilled within twenty-four hours, or partial oxidation will occur. In the distilling-flask a small quantity of the couple should be placed, and also a small piece of 100-mesh copper gauze. This latter determines the precipitation of any minute traces of arsenic which may have escaped elimination by the preceding treatment, and also prevents bumping. The distilled acid is perfectly free from arsenic.

Good commercial muriatic acid may also be used, but in this case the first treatment must be repeated twice, as, owing to the traces of iron chloride and other impurities contained in the acid, the first lot of couple is generally completely dissolved, and does not fully eliminate the arsenic.

The residue in the distilling-flask serves conveniently for the preparation of fresh copper-tin couple, as the proportion in which these two metals are present in the couple is not important.

Discussion.

The President (Mr. Bevan) having invited discussion,

Mr. Hehner desired personally to thank the authors for laying down the conditions under which the sodium treatment was fully successful. As he had explained a few years ago, he had often succeeded in purifying satisfactorily very bad zinc by treatment with sodium, but he had often failed to improve materially even comparatively pure zinc. He had not, however, had so much patience as the authors, and so had never ascertained the exact conditions that were necessary. He was very glad now to find that the authors had brought what was formerly an entirely rule-of-thumb process into something like law and order.

Mr. Chapman said that his own experience was that it was now rarely necessary to purify zinc from arsenic, the great difficulty at the present time being to get zinc that would give out the full amount of arsenic that was put in. Consequently, the problem was not quite so urgent a one as it was when Mr. Hehner took the matter up a few years ago. There could, he thought, be no doubt whatever that the sodium method, if properly applied, was capable of removing the arsenic. He himself had sometimes been unsuccessful with it, but that was probably because he had not known the precise conditions necessary for success. The many experiments he had made, however, went to show that, unless the sodium was entirely removed, the zinc obtained was distinctly "insensitive." He thought that that was proved by the mirrors which the authors had shown. For example, their mirror for the $\frac{1}{100}$ mgm. of arsenic was not more than half that which he was in the habit of getting, and which he had every reason to believe to be correct. The same applied to the other mirrors. Through the kindness of Dr. Thorne, he had had the opportunity of examining some of this sodium-treated zinc with his own materials, and the conclusion he came to was that it was distinctly insensitive. If, therefore, the sodium treatment was to be employed, it must be with very great care, and the operator must recognise the possibility, or even probability, of the resulting zinc being more or less insensitive. The method for the purification of hydrochloric acid recommended by the joint committee of the Society of Chemical Industry and the Society of Public Analysts (treatment with bromine and sulphurous acid and distillation) in some cases worked well, but in others did not. He had, however, for some time been invariably successful in purifying the most impure acid by a slight modification of that method. The only modification was that the strong acid was distilled, instead of being first diluted down to constant boiling-point strength. The pure acid obtained amounted to about three-fifths of the total quantity treated. The only failure he had had was due to the mechanical carrying over of a little iron into the distillate from a very impure acid. This, as had been recently shown, caused diminished sensitiveness, but it was easily obviated by a suitable arrangement of the distilling apparatus.

Dr. DYER said that Mr. Chapman had anticipated a remark that he had been about to make regarding the faintness of the authors' mirrors. Their mirror from $\frac{1}{100}$ mgm. appeared to be less than the mirror obtained in his laboratory from half that quantity of arsenic, working with acid which gave a good blank, and using tubes of approximately the same size as those shown. That seemed to point to slight insensitiveness of the authors' zinc.

Mr. H. D. LAW said that, from the results of the investigation recently communicated to the Society by Mr. Chapman and himself, it was evident that sodium was one of those metals which seriously interfered with the sensitiveness of zinc. He had since made some experiments to find out the particular reducing efficiency of hydrogen liberated by sodium and potassium. By passing an electric current through a solution of sodium hydroxide or potassium hydroxide, a deposit of sodium or potassium was obtained. This dissolved in the water, with liberation of hydrogen at the potential of the metal. The reducing power of this hydrogen had been compared with that of the hydrogen liberated by various metals of different potentials. Camphor was very readily reduced with lead electrodes, but when he tried to reduce camphor with the hydrogen liberated by sodium or potassium, he found that he was quite unable to do so. This showed that the potential of the sodium or potassium was very much lower than that of zinc or lead. He then took different materials which were capable of being readily reduced, such as benzaldehyde, in order to find out, by lowering the reducing potential, at what point a degree of reduction was obtained corresponding with that produced by sodium hydrogen. With lead electrodes benzaldehyde yielded a very sticky mass quite characteristic of high-potential reduction, while with metals of lower potential the sticky masses produced were very much less in quantity. Finally, he took refined copper electrodes, which yielded no sticky substance at all, but a crystalline, brittle hydrobenzoin corresponding to that obtained by reduction in sodium and potassium solutions. This proved conclusively, he thought, that the potential of sodium and potassium was about the same as that of copper, which was extremely low.

Dr. THORNE said that he was inclined to think that, if there was any diminution at all in the sensitiveness of the zinc, it was but very slight. Personally, he was strongly of opinion that in many cases where zinc was said to be highly sensitive either it or the acid was not absolutely free from arsenic. He had certainly not had the same success as Mr. Chapman in obtaining zinc that would give an absolutely clear blank without repurification, though there had undoubtedly been a great improvement in commercial zinc during recent years. As a test of the absolute purity of the zinc, they ran blank determinations for an hour and a half. A twenty-minutes' blank might to a certain extent be satisfactory, but they had found after a large experience that the very small traces of arsenic which were present in most commercial zincs tended to cause irregular results, giving undue prominence to small quantities as compared with larger quantities of arsenic in the material under examination. They found the sensitiveness of each successive batch of zinc purified to be practically the same, and although he was quite prepared to admit that the mirrors they obtained might be slightly less intense than the small-quantity mirrors obtained with other zincs, he thought that the continuous concordance of the mirrors after repeated treatments with sodium showed that they were truly representative, and that the danger of loss of sensitiveness by the treatment was practically nil. The more strongly marked character of the low-quantity mirrors in other cases was probably due, in part at any rate, to traces of arsenic in the other materials. With reference to Mr. Hehner's remarks, he should like to say that a good deal of the patience expended on this matter had been that of his collaborator, Mr. Jeffers. With regard to hydrochloric acid, they also had found that the method first proposed by the joint committee did not answer in all cases. They did not say that the method they had described was a better one than the committee's method as now modified, but it had been found reliable with ordinary impure acid as well as with acid of good quality, and was a simple and convenient method.

In answer to a question put by Dr. Dyer, Dr. Thorne said that they had not tried Mr. Chapman's device of introducing cadmium sulphate into the flask.

Mr. Chapman said that he could put forward in support of his own contention exactly the same argument which Dr. Thorne had used. The mirrors he obtained were perfectly concordant, whatever the method or materials were. Sometimes he put cane-sugar, soluble starch, etc., into the flask, sometimes he left it out; but the mirrors in either case were always of the same intensity within the very narrow limits of experimental error. He asked what evidence there was that the appearance of a bright surface on the zinc coincided with the disappearance of the whole of the sodium.

Mr. Jeffers said that he did not think it could exist alongside the zinc at that temperature.

Mr. Chapman said that it might quite well exist in very small quantity as an alloy dissolved in the zinc.

Mr. Jeffers said there was no room for doubt as to the complete removal of the sodium. If the sodium were not entirely removed, it was impossible to get a bright surface on the zinc at a red heat.

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NOTE ON DUTCH CHEESE.

By CECIL H. CRIBB, B.Sc., F.I.C.

(Read at the Meeting, February 7, 1906.)

In the earlier part of last year I received, amongst a number of other samples of Dutch cheese submitted to me under the Sale of Food and Drugs Acts, some which contained extremely minute proportions of fat. In view of the general recognition of the fact that Dutch cheese is not made, except in the case of certain well-defined brands, from whole milk, it became necessary to consider whether the sale of such samples brought the vendors within the provisions of the Act—*i.e.*, whether they should be regarded as genuine or adulterated.

On looking up the literature of the subject, the question seemed fairly simple. Practically all the published analyses give proportions of fat far exceeding those in the samples I have just referred to, the figures given in every case but one ranging from 19 to 33 per cent. The sole exception to this occurred in a paper by Messrs. Pearmain and Moor (ANALYST, xix., 145), where a fat content of 10·6 per cent. is recorded. I also found that during the last four or five years there have been several prosecutions for selling, either as Dutch cheese or as cheese, samples which were more or less deficient in fat (*British Food Journal*, 1900, p. 47; 1901, p. 202; 1903, pp. 90, 159, 164, 229); and in those cases in which Dutch cheese had been specifically asked for, and in which the proportion of fat was at all comparable with that in the cheeses examined by myself, a penalty was inflicted. I felt justified under these circumstances in certifying adulteration, and proceedings were instituted against the vendors. Counsel appeared for both sides, and after a somewhat protracted trial the magistrate decided that there was no standard, and that the prosecutor had therefore been supplied with cheese which was of the nature, substance, and quality demanded. As during the hearing of some of the cases referred to above, somewhat widely varying opinions had been expressed as to the amount of fat *Dutch* cheese should contain, I thought it desirable to get some idea as to the range of fat content in the Dutch cheese which was actually being imported into the country at the time, and by the kindness of two of the largest firms in the wholesale trade I obtained a series of samples which fairly represent every price and quality coming over at that particular date, the results of analysis of which, together with those of the official samples submitted to me, are given in the following table:

I. FROM WHOLESALE HOUSES.

	Wholesale Price per Cwt.	Approximate Retail Price per Pound.	Fat.	N 6·33.	Ash.	Water.
1. Gouda ...	28s.	4d. to 5d.	1·64	—	5·71	55·22
2. Edam ...	30s.	,,	3·83	25·34	5·81	60·38
3. ,, ...	34s.	,,	4·04	31·92	7·33	53·66
4. Gouda ...	28s.	,,	5·01	28·53	5·29	54·26
5. ,, ...	28s.	,,	5·02	24·89	7·30	58·26
6. ,, ...	32s.	5d. to 6d.	5·36	22·05	5·30	59·28
7. ,, ...	34s.	,,	4·56	34·22	6·10	54·83
8. ,, ...	34s.	,,	4·73	25·34	6·26	60·17
9. Derby shape...	36s.	6d.	4·75	31·92	5·67	54·10
10. Gouda ...	38s.	,,	13·6	23·91	5·48	56·15
11. ,, ...	42s.	,,	12·45	26·67	5·43	50·46
12. ,, ...	44s.	,,	13·2	25·34	4·26	50·63
13. ,, ...	42s.	,,	15·3	23·90	5·15	50·96
14. ,, ...	42s.	,,	18·37	24·54	4·48	52·45
15. Derby ...	46s.	7d. to 8d.	15·95	24·37	5·27	49·45
16. Edam ...	52s.	,,	15·12	24·00	5·54	52·50
17. Edam (full cream) ...	58s.	8d.	24·4	26·05	5·58	41·70
18. "Case" cheese	65s. 4d.	10d. to 1s.	27·9	21·89	4·28	46·2

II. Official Samples.

	Fat.	N 6·33.	Ash.	Water.
19.	1·89	36·24	7·47	56·89
20.	2·75	31·02	8·82	55·40
21.	5·20	31·37	7·15	53·80
22.	2·65	38·46	10·8	51·90
23.	6·62	26·85	6·47	58·80
24.	15·40	—	6·07	50·14
25.	27·12	—	5·94	43·70

The most striking thing about these figures is the vast difference between the proportions of fat occurring in my samples and that found by the various authors to whom I have previously referred. For, excepting Messrs. Pearmain and Moor, all previous observers seem to have found what, judging from my analyses, must be regarded as the maximum figures for fat. It seems clear that a great change had taken place in the composition of Dutch cheese since the earlier analyses were made.

I was informed that the great bulk of the trade is concerned with cheese of the quality represented by the samples numbered 10 to 16, and that at the time the samples were given to me the total bulk of this quality which was sold amounted to fifty times as much as that of the poorer kinds represented by numbers 1 to 9. I was also told that the greater part of the latter was imported into England for one large retail firm, with branches all over the country, who, by selling this inferior article, were gradually forcing their poorer competitors to deal in goods which they would formerly have refused to touch, and thus the whole trade was gradually becoming demoralized.

It is quite obvious from the proportions of fat they contain that the samples numbered 1 to 9 really represent a class of product altogether different from numbers 10 to 16, and are plainly made entirely from separated milk. Considering that the cheeses were valued almost entirely by the unaided senses, it is remarkable how closely the proportions of fat run parallel with the price; and although in many cases a larger percentage of fat was expected than actually turned out to be present, there is no reason to suppose that an expert buyer would be under the slightest delusion as to what he was getting if he came across any of the first nine samples on the list. On the other hand, the ordinary householder would probably fail to see any difference between members of the first and second groups.

As the main point in the defence was the absence of an official standard for cheese, the local authority for whom I acted addressed the Board of Agriculture on the subject, and requested that they would consider the advisability of fixing such a standard. Similar action was at the same time taken by a large number of county and borough councils throughout the country, namely, at the instigation of the Council for the County Borough of Bournemouth.

In response to these communications a letter was received from the Board of Agriculture, saying that "there were difficulties in the way of framing suitable regulations of the nature desired," owing to the existence of so many different varieties of cheese, and that it was not clear in what way such regulations "would assist consumers in obtaining the article they desire." They state that, "on consideration of all the circumstances," they did not feel that "any advantage would be gained by the attempt to deal with cheese by the issue of regulations under the Act."

This decidedly unsatisfactory document entirely fails to answer the question to which I want to direct attention in the present communication—namely, Should the sale of these separated-milk Dutch cheeses be held to constitute adulteration? The question is not merely the academic one of clerical procedure, but involves a matter of much greater importance. If the Board of Agriculture, having power to fix standards, do not use it, their failure to do so is likely to prove a most serious hindrance to the proper administration of the Acts. Before the 1899 Act came into force the magistrate relied almost entirely on the Public Analysts for information as to what constituted adulteration, but now the first question he asks is usually, What is the Government standard? And if there is not one, he in many cases at once concludes that the Government have refrained from fixing one because they were unable, and not because they had never tried, the obvious conclusion being that, if they had failed, no one else could succeed.

It is quite plain that now the reply of the Board of Agriculture has become more or less public property, the analyst who attempts to support a prosecution in the case of a separated-milk Dutch cheese will be in a much more difficult position than he would have been in before that reply was issued. Whatever the law may have to say on the subject, the Public Analyst is bound to report the sample as either genuine or adulterated, as the Act recognises no halfway house between the two. Put quite briefly, I personally think that such samples as those referred to should be returned as adulterated for the following reasons: Dutch cheese has been known for a very large number of years, and its reputation was built up during a period when such products as some of the worst samples were practically unknown, or, if they existed, were openly sold as *skimmed* or *separated* milk cheese. It is used, not as a condiment, but as a cheap and convenient source of nourishment, mainly by the poorer classes, and in probably the greater number of cases without butter being taken at the same time, so that the almost complete absence of fat interferes seriously with its digestibility. Apart altogether from this, the great deficiency of fat carries with it other defects, which are most distinctly to the prejudice of the purchaser. The most important of these is the fact that the presence of so little fat enables the cheese to retain a much larger amount of water without betraying the fact in any way, and as a further result it dries much more rapidly, and in a short time gets so hard that it can only be cut with a hammer and chisel.

The obvious explanation of the poor quality of much of the Dutch cheese now on the market is to be found in the use of separated instead of skimmed milk. As far as my information goes, all the reputable kinds of Dutch cheese were formerly made either from whole milk or from the whole morning milk mixed with the skimmed milk of the previous evening. Now that cheese is mainly the product of a factory instead of a farm, it is not likely that the old and inefficient process of hand-skimming is employed at all, so that even if the old traditions were adhered to as regards the cheeses other than those made from whole milk, there would be a lowering of the fat content to at least the extent shown by my analyses.

As these facts are not known to the public, it seems to me that this is a case, if ever there was one, in which the Board of Agriculture should exercise its powers, especially as the objections it advances to fixing standards are by no means convincing, and, so far as Dutch cheese is concerned, have but little force.

DISCUSSION.

The PRESIDENT (Mr. Bevan), in inviting discussion, said that, personally, he thought that the justice of the case would be met if cheese made from skimmed milk were required to be so labelled.

Dr. VAN RIJN said that, as Mr. Cribb had already very clearly explained, a good deal of cheese had been sent over from Holland lately—during the last year or so—which contained only a very small proportion of fat. The reason for this was that in some parts of Holland, where several years ago no cheese at all used to be made, all the separated milk was now used for making cheese. A market was found for it over there at the price it was worth, but, of course, the people who made it could not be responsible for what was done with it later on. He personally thought that it was not fair to sell this "separated cheese," as he would call it, simply under the name of Dutch cheese. It was, however, very difficult—he thought almost impossible—to fix a legal standard for cheese generally. Something in that direction had been attempted in Holland, and now combinations of cheesemakers were being formed for the purpose of doing what had been done with regard to butter control—namely, to fix a standard for themselves and to establish stamps or marks for their cheeses. For instance, it would not be permissible to make a cheese of the ordinary Gouda or Edam shape containing less than about 15 per cent. of fat (25 per cent. fat in the dry residue). He thought that the majority of the cheesemakers at the moment were in favour of such a limit. It would not have any legal force, but would be a commercial standard. If it were adopted it would naturally strengthen greatly the position of the authorities in such prosecutions as had been lately instituted in this country. He should like to explain why so low a limit as about 15 per cent. was suggested. For Edam cheese it was the custom to mix the evening milk, after the cream had been taken off, with the full-cream morning milk, and to make the cheese from this mixture, which contained, especially at some seasons of the year, a somewhat low proportion of fat. From experiments made with normal milk of varying degrees of richness at a Government station in Holland, the lowest proportion of fat in cheese made in this way was found to be 20 per cent. The lower limit of 15 per cent. was proposed in order that a margin might be allowed to cover possible abnormalities in the milk used. It was, moreover, possible to make good cheese containing this amount of fat. With a much smaller quantity of fat the quality of the cheese was quite different. He did not know much about the digestibility of cheese containing only a few per cent. of fat, but it certainly was not a first-class commercial product.

The question of the estimation of fat in cheese had been already mentioned in the ANALYST about a year ago. He had just had an opportunity of discussing it with one or two colleagues, whose opinion had been that the same results would be obtained whichever of the ordinary methods might be used. This was, practically speaking, correct in the case of full-cream cheese containing from 30 to 40 per cent. of fat, but it had been stated very clearly in Holland by several analysts that, with cheese containing a low percentage of fat (below 22 per cent.), the method of extraction with ether always yielded 2 or 3 per cent. less fat than was actually present. He might perhaps later on have something to add in reference to this.

Mr. HEHNER said that many years ago he had called attention to this problem, because it seemed to him very anomalous that, while a man was allowed to sell cheese made from milk from which the fat had been taken, as soon as he tried to confer a benefit on the purchaser by filling up the cheese with other fat he was punished. He thought it unreasonable to punish the vendor of margarine cheese while leaving the vendor of skimmed-milk cheese free. He had never found any difficulty in distinguishing by taste and appearance between skimmed-milk cheese and full-cream cheese, but a "filled" cheese was very difficult to distinguish without chemical analysis. At one time cheese might fairly be considered to be, roughly, one-third water, one-third fat, and one-third other things. But times had since changed considerably. Cheese was now one of those manufactured products which allowed the maker a good deal of scope; and the courts had laid down—he thought rightly—that the manufacturer had a right to exercise his ingenuity in the manufacture and composition of a manufactured product. A food material that was virtually sold as Nature furnished it must be as Nature gave it, but as soon as the question of manufacture came in there must necessarily be a certain latitude. That was the case with cheese and with many other things. It was, however, very difficult in a case like that of cheese to give a notice consistently. It was imaginable that boxes should be branded, but it was almost impossible to mark each piece of cheese sold. If, however, some means could be introduced for distinguishing at once—say, by colour—between full-cream cheese and skimmed-milk cheese it would probably be a boon to the consumer. But the Dutch manufacturers were very scientific, and understood very well how to sell both butter and cheese at the same time at a good price, and it would be much better that they should, in the way that Dr. van Rijn had mentioned, themselves take steps to guard against the mischief resulting from too acute competition. He thought that everybody must agree with Sir Thomas Elliott that it was exceedingly difficult to lay down a general law. Several kinds of cheese had, by common consent, been allowed to be made from skimmed milk—for example, Parmesan, Dutch, and some Dorset cheeses—and, after all, what one person had the right to do could not well be denied to another. In fact, he had gradually come to consider that cheese was now one of those products which were entirely manufactured, and that the question was one in which the public and not the analyst must be the final arbiter.

Mr. JOHN WHITE thought that all that Mr. Cribb wanted was what he himself had wanted when he was concerned in a similar campaign which had resulted disastrously—namely, not to stop the sale of the article, but to let it be sold for what it was, viz., skim-milk cheese. It was right that the skill of the manufacturer should be taken into account, provided that the article made was being improved; but here the skill of the manufacturer had been employed to produce, not an improved article, but one of diminished value. With regard to the position of the public as the ultimate arbiter, he could not conceive that anybody would buy this stuff a second time. After being kept for two or three days it became exceedingly hard. Personally, he considered it a gross fraud to sell as cheese, or as Dutch cheese, an article containing only 1·6 per cent. of fat. As to the estimation of the fat, he had found that such small quantities could only be accurately determined by what was practically the Werner-Schmidt process—namely, by boiling with water and hydrochloric acid, cooling, and shaking out with ether.

Mr. Fisher drew attention to the results shown by a sample purchased as Dutch cheese, which he had analysed in 1903. It contained only 1·6 per cent. of fat, with 57·1 per cent. of water and the vendor was in consequence fined £10.

Mr. Cribb, in reply, said that he had adopted practically the same method of fat estimation as Mr. White. He could confirm what Dr. van Rijn had said as to direct extraction with ether. When the quantity of fat was large the error was, for practical purposes, of no consequence, but with a small percentage of fat the error, which was still about the same in absolute weight, bore a larger proportion to the total. It seemed to him that if the manufacturer of cheese were to have an entirely free hand he might make an article containing nothing but casein, water, and a little mineral matter, but nobody would pretend that to the consumer that was Dutch cheese as ordinarily understood. As to the suggested difficulty of notification, he could not see any difference between the case of cheese and that of butter and margarine, with regard to which provisions as to labelling were already in existence. If the Sale of Food and Drugs Acts were carried out properly, any radical change in a well-known article of food would be discovered at once. Under the present conditions any such change was, as a rule, not found out until it had been so long in operation as to become an established trade custom, which could not be altered without inconvenience and loss to many. All this pointed to the necessity for a more thorough application of the Sale of Food and Drugs Acts, and for the taking of much larger numbers of samples than were taken November 22, 1906.

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ORDINARY MEETING OF THE SOCIETY.

This was held on Wednesday evening, March 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. G. Craig, J. B. Gall, H. N. Hanson, B. W. Methley, F. D. Ratcliff, and F. Robertson, were read for the second time; and certificates in favour of Messrs. Ernest Quant, 2, Park

Crescent, Torquay, analytical chemist; Frederick Wilson Montrose Ross, 21, Soho Square, London, W., analyst to Messrs. Crosse and Blackwell, Ltd.; and Frank Ernest Thompson, A.R.C.Sc., A.I.C., the Laboratory, Walsall, analytical and consulting chemist, were read for the first time.

Messrs. S. Dickson, J. Evans, F. Hughes, G. Patterson, and H. Thompson, were elected members of the Society.

The following papers were read: "A Simple and Rapid Method for the approximate Estimation of Boric Acid," by Cecil H. Cribb, B.Sc., and F. W. F. Arnaud; "Analysis of a Sample of Air extinguishing Flame," by Bertram Blount; "The Detection of Cocoanut Oil in Butter," by Arthur W. Thorp; and "The Composition of Milk," by H. Droop Richmond.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A New Centrifugal Method for Determining Fat in Milk—Wendler's Method. Von Küttner and Ulrich. (*Zeit. offentl. Chem.*, 1906, xii., 41-58.)—The process consists in mixing the milk with a saline solution, adding a little isobutyl alcohol, and subjecting the mixture to centrifugal action, the procedure being similar to that of Gerber's method. The saline solution employed contains tartrates, sodium chloride, and sodium hydroxide (proportions not given). Eleven c.c. of this solution, 10 c.c. of the milk to be tested, and 0·6 c.c. of isobutyl alcohol are successively introduced into a butyrometer tube and the latter then closed with an indiarubber stopper. The contents of the tube are mixed, the tube is immersed for two or three minutes in water at a temperature of 45° C., and then submitted to centrifugal action in the usual way. The surface separating the fat from the saline solution is quite horizontal, rendering the reading of the volume of the fat an easy matter. If the contents of the tube have been well mixed, no flocculent layer ever occurs between the fatty and aqueous layers. The results obtained, as shown by 360 comparative determinations, agree with those yielded by Gerber's method. W. P. S.

A Rapid Method for the Detection of Traces of Zinc in Wort, Beer, Wine, etc. J. Brand. (*Ztschr. ges Brauw.*, 1905, xxviii., 440; through *Chem. Zeit. Rep.*, 1906, xxx., 25.)—The liquid to be examined is acidulated with hydrochloric acid, and a few drops of potassium ferrocyanide solution are added. The presence of even a trace of zinc will cause a flocculent precipitate of zinc ferrocyanide to be produced, which, being mixed with much albumin, settles rapidly, and may be readily filtered after decanting the bulk of the liquid. The washed precipitate is ignited, and the residue treated with acetic acid; in the solution obtained zinc is identified with hydrogen sulphide. As little as 1 part of zinc in 500,000 parts of beer, etc., can be readily detected by this method. A. G. L.

Hordenine: A New Alkaloid extracted from the Germ of Barley. E. Léger. (*Journ. Pharm. Chim.*, 1906, xxiii., 177-181.)—A crystalline alkaloid has been separated by the author from the dried germs of malted barley. It was first obtained in ethereal solution by means of Stas' method, and the residue left on evaporation purified by repeated crystallization from alcohol and decolorized with animal charcoal. The yield of *hordenine* appears to vary with the temperature at which the malt was dried, and the author has obtained from 0·22 to 0·45 or 0·5 per cent. from different germs. The alkaloid crystallizes in orthorhombic prisms, which show double refraction. They are anhydrous, colourless, and almost tasteless, and melt at 117·8° C. (corr.) to a colourless liquid. Hordenine slowly sublimes at its melting-point and rapidly at 140° to 150°. It is readily soluble in alcohol, chloroform, and ether, less soluble in benzene, only slightly soluble in water, and almost insoluble in cold petroleum hydrocarbons. It dissolves easily in hot carbon tetrachloride, but separates out almost quantitatively on cooling. It acts as a strong base, giving a red coloration with phenolphthalein and displacing ammonium from its salts in the cold. It gives no coloration with strong sulphuric acid, and is hardly attacked by a hot concentrated solution of potassium hydroxide. On the other hand, it reduces potassium permanganate in acid solution, hot silver nitrate solution, and iodic acid solution, yielding a precipitate of iodine. Its formula, calculated from the composition of its salts, is $C_{10}H_{15}NO$. It is a tertiary monoacid base, and thus forms only a single series of salts, all of which are very soluble in water. C. A. M.

Determination of Chloral Hydrate. T. E. Wallis. (*Pharm. Journ.*, 1906, vol. 76, 162, 163.)—The method proposed consists in heating the chloral hydrate with sodium hydroxide in alcoholic solution and titrating the sodium chloride formed. The determination of the amount of sodium hydroxide absorbed gives untrustworthy results (*cf.* ANALYST, 1903, xxviii., 189), unless strict conditions be adhered to. The details of the method are as follows: A weighed portion of from 0·1 to 0·4 gram of the chloral hydrate is dissolved in about 10 c.c. of the alcohol, and introduced into a thick-walled bottle. A measured volume of $\frac{N}{1}$ sodium hydroxide solution is added, the bottle is closed by an indiarubber stopper, which is tied down, and the bottle with its contents heated for three hours in a water-bath. After cooling, the solution is neutralized with sulphuric acid, using phenolphthalein as indicator, and then titrated with $\frac{N}{10}$ silver nitrate solution. The reaction is represented by the equation,

$$CCl_3CH(OH)_2 + 5NaOH = 3NaCl + 2HCOONa + 3H_2O.$$

It is important that the contents of the bottle during the heating contain at least 50 per cent. of alcohol by volume. W. P. S.

The Determination of Traces of Chloroform. M. Nicloux. (*Comptes Rendus*, 1906, cxlii., 163-165.)—Chloroform when present in alcoholic solution in a quantity not exceeding 0·1 gram can be determined by diluting the liquid to 60 c.c. with alcohol, and boiling it for an hour under a reflux condenser with 10 c.c. of a 10 per cent. alcoholic solution of pure potassium hydroxide. When cold, the contents of the flask are mixed with 15 c.c. of water, exactly neutralized with phenolphthalein as indicator, and treated with potassium chromate, and the chlorine titrated with standard silver nitrate solution (8·535 grams per litre), each c.c. required representing 2 mgms. of chloroform. Air containing chloroform is analysed by being drawn through alcohol (95 per cent. strength), contained in two absorption vessels, at the rate of 1 litre in half an hour, and the absorbed chloroform is then determined as above described. In the case of blood and aqueous liquids it is necessary to add strong alcohol (80 to 95 per cent.), acidified with 0·25 gram of tartaric acid in the proportion of 5 parts to 1 of the sample, and to distil the mixture in a fractionating flask until about a third has passed over. The distillate is received in 10 c.c. of 95 per cent. alcohol, and the chloroform determined as before. The results thus obtained are invariably 1·5 to 2 per cent. too low. C. A. M.

The Constituents of Myrrh. A. Tschirch and W. Bergmann. (*Archiv. Pharm.*, ccxliii., 641; through *Pharm. Journ.*, 1906, vol. 76, 128.)—Picked Somali myrrh was found to have the following composition: Soluble in alcohol (resin and oil), 28 to 30 per cent.; insoluble in alcohol (gum and enzyme), 61 per cent.; impurities, 3 to 4 per cent.; moisture, 5 per cent.

Part of the substances soluble in alcohol was insoluble in ether, the remainder (about 21 per cent. of the myrrh) being soluble. The portion insoluble in ether was redissolved in alcohol, and separated by precipitation with lead acetate into α-heerabo-myrrholol and β-heerabo-myrrholol, the latter remaining in solution. These substances were obtained in the form of yellowish-brown and grayish-brown amorphous powders respectively.

The portion of the alcoholic extract soluble in ether was dissolved in this solvent, and treated with potassium hydroxide to remove α-and β-heerabo-myrrhol, which were afterwards separated by means of lead acetate, when they were obtained as grayish-yellow powders. The ethereal solution was next evaporated and distilled in a current of steam. By this means a residue of heerabo-resene was obtained in the retort, whilst the distillate consisted of yellow oil. The latter had a specific gravity of 1·046, rapidly resinified, and bore no relation to the resin acids, resinotannols, resinols, etc., found in other resins.

The substances insoluble in alcohol, consisting of the gum and enzyme, could not be separated from one another. The gum yielded arabinose on hydrolysis. In addition to these substances, myrrh also contains a bitter principle, but all attempts to isolate it failed. W. P. S.

Determination of Cinnamic Acid in Storax. D. Hooper. (*Pharm. Journ.*, 1906, vol. 76, 107.)—The determination of the amount of cinnamic acid in storax is proposed as a method of detecting the presence of adulterants in this balsam.

Tschirch and Itallie have shown that true Oriental storax contains 23 per cent. of cinnamic acid, and other investigators have concluded that the percentage of the acid should not fall below 20. A weighed quantity of the storax is saponified in alcoholic solution with potassium hydroxide, evaporated to remove the alcohol, the residue dissolved in water, and the solution shaken out with ether to remove unsaponifiable substances. The solution is then treated with sulphuric acid in excess, and the voluminous precipitate collected on a filter and washed. The precipitate is next dissolved in hot water, and the crystals which separate on cooling are collected, dried, and weighed. W. P. S.

Adulterated Santal-Wood Oil. E. J. Parry. (*Chemist and Druggist*, 1906, lxviii., 211.)—The low price of large parcels of this oil imported from Germany caused suspicions to arise as to the purity of the oil, and the author has recently examined a sample which enabled him to definitely decide the nature of at least one of the adulterants present. The oil had a specific gravity of 0·970 at 15° C., an optical rotation of $-14°$ in a 100 mm. tube, was soluble in 5 volumes of 70 per cent. alcohol, and had an apparent santalol value (free) of 93·5. By repeated fractionation under reduced pressure a small amount (about 5 per cent.) was obtained, which boiled at 220° C. at ordinary pressure. The specific gravity of this fraction was 0·943, and the refractive index 1·4823 at 20° C. The odour left no doubt as to its being terpineol. This was confirmed by the melting-point (115° C.) of the nitrosochloride yielded by the fraction. W. P. S.

The Purity of Balsam of Peru. Utz. (*Pharm. Post.*, xxxix., 21; through *Pharm. Journ.*, vol. 76, 129.)—Adulteration of this balsam is, according to the author, the rule rather than the exception. Turpentine and gurjun balsam appear to be the favourite adulterants, and the usual tests often fail to detect them. Such adulterated balsams may show a cinnamein content of 50 to 60 per cent. The physical characters of the cinnamein obtained sometimes indicate sophistication. It should be brown and homogeneous, whereas with turpentine it is often granular or even solid. The refractive index gives more satisfactory results, as for pure balsam the value varies from 1·5862 to 1·5878 at 15° C.; whilst the refractive index of gurjun balsam is 1·5142; of castor oil, 1·4809; and of paraffin oil, 1·4821. On the other hand, perugen, which is a factitious substitute for balsam of Peru, possesses a refractive index of 1·5863. Perugen, however, gives an intense olive-green coloration with Cæsar and Loretz's nitric acid test, instead of the golden yellow yielded by pure balsam of Peru. W. P. S.

TOXICOLOGICAL ANALYSIS.

Toxicity of Cyanogen Compounds towards Fish. J. Hasenbaümer. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, 97-101.)—The author has carried out a considerable number of experiments regarding the action on fish of certain cyanogen compounds when the latter were added to the water in which the fish lived. The investigation was in connection with an inquiry regarding the discharge of effluent containing traces of these compounds. Potassium cyanide was found to be extremely poisonous to fish, 0·0018 gram per litre of water killing tench and goldfish in a very short time The following quantities per litre of other cyanogen compounds also killed fish of these species: Potassium ferrocyanide, from 1·5 to 3·0 grams; potassium ferricyanide, from about 1·7 gram; potassium or ammonium thiocyanate, about 1·5 gram.

W. P. S.

ORGANIC ANALYSIS.

Temperature of Combustion of Methane in presence of Palladiumized Asbestos. H. J. Denham. (*Journ. Soc. Chem. Ind.*, 1905, xxiv., 1202.)—An investigation undertaken to ascertain to what extent methane is burnt when passed over palladiumized asbestos in presence of oxygen has shown that (1) the temperature of combustion of a mixture of 1 volume of methane with 2 volumes of oxygen lies between 514° and 546°; (2) an increase in the speed at which the gases are passed through the tube causes a decided increase in the temperature of combustion; (3) a variation in the volumes of the gases influences the temperature of combustion, tending to raise it beyond the above-mentioned range; and (4) the addition of hydrogen, even in large quantities, does not cause the methane to burn at a lower temperature. It is concluded, therefore, that the usual method of fractional combustion of hydrogen gives reliable results, if the temperature does not exceed 500° to 550°. The catalytic action of the metal does not appear to be due to superficial oxidation, and in no case has anything approaching complete combustion been observed, even when the gas was 100° above its combustion temperature.

W. H. S.

The Volumetric Determination of Pentoses. A. Jolles. (*Berichte*, 1906, xxxix., 96, 97.)—From 0·2 to 1·0 gram of the substance under examination is treated with 200 c.c. of hydrochloric acid (specific gravity 1·06), and distilled in a current of steam until Bial's reagent shows that no more furfural is formed, an additional 100 c.c. of the hydrochloric acid being introduced into the flask during the distillation. The furfural is now determined in an aliquot part of the distillate by treating it, after neutralization, with a known excess of standard potassium bisulphite solution, and titrating the excess with standard iodine solution after the mixture has stood for two hours:

$$C_4H_3O.COH + HKSO_3 = C_4H_3O.CH(OH)SO_3K.$$

Each c.c. of normal bisulphite solution is equivalent to 0·07505 gram of pentose.

C. A. M.

The Detection of Olive Oil extracted by Means of Carbon Bisulphide in Soap. J. Vamvakas. (*Ann. de Chim. Anal.*, 1906, vol. 11, 53, 54.)—The soap manufactured in Crete is prepared almost exclusively from olive oil, and the author has long used the following simple method for determining whether the original oil had been extracted by means of carbon bisulphide: 2 grams of the soap are dissolved in 100 c.c. of 95 per cent. alcohol, the solution filtered, and its colour noted. If colourless or gray the oil was obtained by mechanical expression, whilst a yellow colour (with green fluorescence) indicates soap prepared from an extracted oil. The addition of a green or yellow dye-stuff, soluble in alcohol, to the soap is stated not to interfere with the test, since in that case the green fluorescence of the solution is even more pronounced (*cf.* ANALYST, xxx., 313). C. A. M.

On the Behaviour of the most Important Vegetable Oils towards Polarized Light. M. A. Rakusin. (*Chem. Ztg.*, 1906, xxx., 143.)—The author suggests that animal and vegetable oils may be distinguished from mineral oils by means of polarized light, and has compiled the following table:

	Colour, etc.	Specific Gravity.	° Ventzke with a Tube of Mm.			Analyst.*	Remarks.
				100	50		
A. LIQUID OILS.							
1. *Non-drying Oils.*							
Olive oil (*Ol. olivarum*)	Yellow.	0·9126 (21° C.)	+ 0·6			R.	Dark, with 200 mm. tube.
,, (church use) ...	Yellowish-green.	0·9120 (21° C.)	+ 0·2				
,, (gallipoli) ...	,,	0·9140 (18° C.)	Opaque.				
,, ...	,,	0·9134 (18° C.)	+ 0·2				
Earth-nut oil	—	—	- 0·4	—	—	B.	
,, ...	—	—	- 0·31 to + 1·15	—	—	C. L.	-7′ to +24′, with a Laurent.
Croton oil	Dark yellow.	0·9389 (19° C.)	+14·5	—	+3·6	R.	Benzene is without influence.
,, ...		0·9384 (18° C.)	—	+8·2	+4·1		
,, ...	—	—	+43	—	—	P.	
Almond oil (sweet) ...	Light yellow.	0·9160 (20° C.)	- 0·1	—	—	R.	
,, (Persian) ...		0·9130 (23° C.)	< - 0·1	—	—		
,, ...	—	—	- 0·7	—	—	B.	
Olive oil	—	—	+ 0·6	—	—	B.	
,, (Provence oil)	Light yellow.	0·9130 (20° C.)	+ 0·2	—	—	R.	Huil d'olive surfine.
,, ...		0·9120 (21° C.)	+ 0·8	—	—	R.	
Rape oil (French) ...	—	—	- 2·1	—	—	B.	
(Japanese) ...	—	—	- 1·6	—	—	B.	
			- 0·23 to - 0·46	—	—	C. L.	-5′ to -10′ with a Laurent.
	Light yellow.	0·9136 (15° C.)	- 0·2			R.	
(*Ol. raparum*)		0·9106 (21° C.)	- 0·1	—	—		
(refined) ...		0·9108 (18° C.)	- 0·2	—	—		Belgian Co.,
Castor oil (Italian) ...	Colourless.	0·9604 (20° C.)	+ 8·0	—	—	R.	
,, ...		0·9596 (18° C.)	+ 8·4	—	—		
,, ...	—	—	< + 8·65	—	—	W.	
,, ...	—	—	+ 40·7	—	—	P.	
Mustard oil (Sarepta) ...	Dark yellow.	0·9161 (19° C.)	- 0·5	- 0·25	—	R.	J. K. Glitschin, Sarepta.
,, (black) ...	—	0·9144 (22° C.)	Opaque.	- 0·1	—		
Sesame oil (cold pressed)	—	—	+ 3·1	—		B.	
,, (hot pressed)	—	—	+ 7·2	—			
,, 1878 ...	—	—	+ 4·6	—			
,, 1882 ...	—	—	+ 3·9	—			
,, 1882 ...	—	—	+ 9·0	—			
,, (Indian) ...	—	—	+ 7·7				
,, (*Ol. sesame Gallicum*)	Light yellow.	0·9247 (14° C.)	+ 1·9			R.	
,, II.		0·9250 (15° C.)	+ 2·4				
,, ...		0·9206 (20° O.)	+ 2·3				
Mustard oil (white).	—	—	- 0·43			C. L.	-9′ with a Laurent.

* B., Bischop; C. L., Crossley and Le Sueur; R., Rakusin; W., Walden.

Oil.	Colour, etc.	Specific Gravity.	* Ventzke with a Tube of Mm.			Analyst.	Remarks.
			200	100	50		
A. LIQUID OILS (*continued*).							
2. *Drying Oils.*							
Cotton-seed oil ...	Light yellow.	0·9220 (22° C.)	− 0·1	—	—	R.	Cotton oil (*Ol. Gossypii*).
Garden cress oil ...	—	—	0	—	—	C. L.	Inactive oil.
Hemp-seed oil (raw) ...	Dark green.	0·9256 (22° C.)	Opaque.	—	+ 0·1	R.	
Linseed oil (raw) ...	Dark yellow.	0·9314 (21° C.)	,,	+ 0·2	—	R.	Filtered.
,, ...	—	—	− 0·3	—	—	B.	
,, ...	—	—	+ 0·28	—	—	C. L.	+ 6′ with a Laurent.
Cameline oil	Dark yellow.	0·9230 (21° C.)	− 0·1	—	—	R.	
Poppy-seed oil	—	—	0 to 0·17	—	—	C. L.	0° to + 4 with a Laurent.
,, ,, ...	—	—	0	—	—	B.	Inactive.
,, ,, ...	Light yellow.	0·9219 (20° C.)	+ 0·1	—	—	R.	
Niger-seed oil	—	—	0 to 0·86	—	—	C. L.	0° to + 18′ with a Laurent.
Walnut oil	Almost colourless.	0·9238 (18° C.)	+ 0·15	—	—	R.	
,, ...	—	—	− 0·3	—	—	B.	
"Safflor" oil	—	—	+ 0·17 to 0·66	—	—	C. L.	+ 4′ to + 14′ with a Laurent.
Sunflower oil	Light yellow.	0·9195 (22° C.)	+ 0·1	—	—	R.	
B. SEMI-SOLID AND SOLID OILS.							
1. *Viscous Oils.*							
Cocoanut oil	White and viscous.	0·9264 (20° C.)	Not examined.		—	—	Crystalline structure.
Cocoanut oil in 50 per cent. benzene solution.	Colourless.	—	+ 0·19 × 2	—	—	R.	
Palm oil	Yellow paste.	0·9508 (18° C.)	Not examined.				
Palm oil in 50 per cent. benzene solution.	Light yellow.	—	0·1 × 2	—	—	R.	
2. *Solid Oils.*							
Cocao butter	Pale yellow.	0·9702 (20° C.)	Not examined.				
Cocao butter in 50 per cent. benzene solution.	Light yellow.	—	0·1 × 2	—	—	R.	
OTHER OILS.							
Laurel oil	Green paste.	0·9335 (19° C.)	Not examined.				
Laurel oil in 25 per cent. ether solution.	Green.	—	—	—	0·9 (× 16)	R.	Tasteless.
Jasmine oil (*Ol. jasmini pingue*).	Almost colourless.	0·9054 (18° C.)	+ 0·9	—	—	R.	
Wild seed oil (*Ol. resedæ pingue*).	Yellow.	0·9122 (18° C.)	+ 0·6	—	—	R.	

A. G. L.

Detection of Artificial Colouring Matter in Fats. A. Olig and J. Tillmans. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, 94.)—If, during the rendering of a fat, the proteid matter be charred through over-heating, the fat becomes more or less tinted, and imparts a yellow colour to the acetic acid used in the test described by Sprinkmeyer and Wagner (*cf.* ANALYST, 1905, 244). In case a coloration is obtained, the fat should be extracted with alcohol (which is also coloured by the charred proteid matter), and the solution further tested for the presence of coal-tar dyes, etc.

W. P. S.

On the Detection and Determination of Paraffin Wax in Mixtures with Ceresin. F. Ulzer and F. Sommer. (*Chem. Zeitg.*, 1906, xxx., 142.)—Various methods of examining mixtures of paraffin wax and ceresin are discussed. The best appears to be the determination of the refractive index at 90° C., the refractive index of paraffin wax with solidifying points varying from 50° to 60° C. lying between 2·0 and 4·0, whilst the values for ceresins solidifying between 68° C. and 71° C. vary from 11·5 to 13. A sample of German paraffin, however, with a solidifying point of 38° C. gave the high refractive index of 6·81. If only a small quantity of paraffin is present, it may be concentrated by boiling 5 grams of the sample for ten minutes with 100 c.c. of 95 per cent. alcohol, filtering hot, and evaporating, the refractive index of the residue obtained being compared with that of the original. Conversely, if only a small quantity of ceresin is present, 5 grams of the sample are dissolved in 50 c.c. of carbon bisulphide, and 100 c.c. of ether are added. Alcohol is then slowly added, the temperature being kept at 25° C. until a copious precipitate is obtained, which is washed with alcohol and ether, dried, and examined for ceresin. Good indications are also obtained by using Crismer and Motton's method of determining the critical temperature of solution in various solvents, ethyl alcohol being preferred. The critical temperature of solution of various paraffins was found to be, in ethyl alcohol, 155·5° to 158; amyl alcohol, 47° to 54°; acetone, 67·5° to 77°; and acetic anhydride, 163° to 176°; whilst some samples of ceresin gave the values 68° to 71°, 174° to 177°, 73·5° to 76°, 95° to 98°, and 191° for the same solvents. A differentiation may also be made by capillarity, ceresin rising much less in filter-paper than does paraffin (6 cm. as against 10 cm. in one hour at 100° C. in the case of two samples compared).

A. G. L.

Determination of Fatty Acids in Textile Soap. Gerhard Krüger. (*Chem. Zeit.*, 1906, xxx., 123.)—Ten grams of the cut-up soap are weighed out into a tared porcelain crucible of 150 c.c. capacity, dissolved in water, and heated with 20 c.c. of dilute (1 : 10) sulphuric acid on a water-bath until the fatty acids have become clear. From 5 to 10 grams of beeswax are then stirred into the fatty acids, and the whole allowed to cool. The solidified fatty cake is then lifted out of the crucible, the acid is poured out, and the cake treated with successive quantities of water until the latter ceases to show an acid reaction. The crucible and fat are then dried for one hour at 70° C., moistened with alcohol and dried for another hour at 100° C., after which they are weighed. The error due to any fatty acid passing into solution in the acid liquid may be neglected, since even for palm-nut oil soaps it is quite small, whilst cocoanut oil is not used for making textile soaps. A. G. L.

Note on Commercial Oil of Turpentine. W. Vaubel. (*Zeit. öffentl. Chem.*, 1905, xi., 429-432.)—The author recommends the determination of the bromine absorbing power as a means of distinguishing between genuine oil of turpentine and its adulterants. American oil of turpentine was found to have a bromine value of 110 to 115; Russian, 72 to 97; resin "essence" (a product of the distillation of resin), 89. The figures express the grams of bromine required per 100 grams of oil. Russian oil of turpentine is distilled from the roots of the trees, and is an unsatisfactory substitute for the genuine oil. W. P. S.

The Physical Characteristics of Certain Copals. Bottler. (*Chem. Rev. Fett u. Harz. Ind.*, 1906, xiii., 1-5.)—A comparative examination has been made of the characteristics of all the more important copals met with in commerce, many of the results being in tabular form. The physical properties included the appearance of the surface, the colour, transparency, lustre, appearance on fracture, specific gravity, hardness, fusibility and solubility. The specific gravities of the copals in most demand ranged from 1·03 to 1·07 for the substance containing air-bubbles, a higher value being obtained after removal of the air. Speaking generally, the smaller the difference between the densities determined before and after removal of the air, the more valuable the copal. Thus the following differences were observed: Lindi copal, 0·001; Zanzibar copal, 0·0015; red Angola copal, 0·014; Brazil copal, 0·014; Cameroon copal, 0·015; Manila copal, 0·059; Hymenæa (South-American) copal, 0·0615; and Kauri copal, 0·064. In the author's opinion the Brazil copal is really a West African and not an American copal. The degree of hardness is an important factor for distinguishing between the different kinds of copals. The hardest is Zanzibar copal, and the other important kinds can be ranged in the following scale: *Hard*—Mozambique, Lindi, red Angola, Sierra Leone (Kiesel), yellow Benguela, white Benguela, Cameroon, and Congo copals; *Medium*—Manila, white Angola, and Kauri copals; *Soft*—Sierra Leone (new), Hymenæa, and Brazil copals.

The melting-points of copals range between 90° and 360° C. The following results were obtained by the author: Hymenæa, 95° C.; Cameroon, 108° C.; Manila, 112° C.; Manila (yellow, hard), 135° C.; Kauri, 126° C.; Kauri, 140° C.; yellow Benguela, 170° C.; white Benguela, 175° C.; Congo, 180° C.; Sierra Leone, 185° C.; Kiesel, 220° C.; white Angola, 245° C.; Lindi, 246° C.; Zanzibar, (*a*) 259° C., (*b*) 265° C.; and red Angola, 305° C. The solvents tried included chlorhydrin, terpineol, and carbon tetrachloride, in addition to the older solvents. A rapid method of dissolving copals (*e.g.*, West African varieties) consists in heating them for forty-eight hours at 100° C., mixing them with sand or glass powder, digesting the mixture for twenty-four hours with anhydrous ether, and finally adding an equal quantity of hot alcohol. Turpentine oil has usually only a solvent action on copals when it contains ozone. The different copals can be arranged in the following order as regards their solubility in the solvents most commonly employed: White Angola, Manila, Kauri, Brazil, Congo, Sierra Leone, yellow Benguela, red Angola, white Benguela, Kiesel, Cameroon, Lindi, and Zanzibar copals. C. A. M.

Recovery of Iodine from the Residues obtained in Hübl's Method.

A. Olig and J. Tillmans. (*Zeits. Untersuch. Nahr. Genussm.*, 1906, vol. 11, 95-97.) —The solutions obtained after the final titration in determinations of the iodine value of fats by Hübl's method are collected until a good quantity is obtained. After removing the chloroform by means of a separating funnel, the aqueous portion is rendered strongly alkaline with sodium hydroxide, and evaporated as far as possible. The mercury compounds (oxide, basic carbonate, and some metal) are filtered off, washed, and the filtrate again evaporated to dryness and ignited. A little mercury will still be present, and care must be taken during the ignition. The residue is dissolved in water, filtered from any insoluble matter, and the solution then rendered strongly acid with hydrochloric acid. The mixture is now warmed, and a concentrated potassium bichromate solution is added until all the iodine has been precipitated. The liquid is then decanted, the iodine is washed with a small quantity of water, and distilled from a retort in a current of steam. The distilled iodine is collected in a well-cooled receiver, from which it may be readily removed in its wet state, and then dried or sublimed. W. P. S.

Notes on the Determination of Calorific Values by means of the Bomb.

D. Aufhäuser. (*Zeits. angew. Chem.*, 1906, xix., 89.)—In order to make the temperature corrections as small as possible, the water used should be initially as much below the normal temperature as it is finally above it. The temperature of the water in the calorimeter may be most conveniently adjusted for this purpose by putting colder water into the calorimeter, placing the bomb in position, starting the stirrer, and then warming the whole to the required temperature by means of a small electric heater, preferably a cylindrical electrical lamp. The lamp should be wetted before it is plunged into the water in order to obviate the error due to adhering water when it is taken out. A. G. L.

The Volumetric Determination of Organic Hydrosulphides and Thio Acids.

P. Klason and T. Carlson. (*Berichte*, 1906, xxxix., 738-742.)—Titration of organic hydrosulphides with an aqueous solution of iodine gives under suitable conditions quantitative results in accordance with the equation:

$$2R.SH + I_2 = R_2S_2 + 2HI.$$

Thiocyanic acid, however, does not react in this way. The presence of bicarbonate is not only unnecessary, but may lead to further oxidation. The weighed quantity of the hydrosulphide is dissolved in alcohol and titrated with N-iodine solution until a faint yellow colour appears. It is essential that there shall be sufficient alcohol present to keep the bisulphide formed in solution, since otherwise it too can absorb iodine, and the results will be too high. The results of test experiments with different classes of thio compounds, such as *p*-thiocresol, methyl and ethyl hydrosulphides, thioglycollic, and thioacetic acid, show that the method is capable of great accuracy. Rosenheim and Davidson found that the reaction between thioglycollic acid and iodine in sodium bicarbonate solution gave a quantitative yield of dithioglycollic acid; but the author finds that the results thus obtained are not constant, and are invariably too high owing to further oxidation with the formation of thioacetic acid. This latter acid, too, also undergoes further oxidation in a bicarbonate solution, acetylbisulphide being produced.

Aromatic hydrosulphides—*e.g.*, thiophenol, β-thionaphthol, and *p*-thiocresol—are so strongly acid that they form salts with alkalies in neutral solutions, and can therefore be titrated with standard alkali solution, phenolphthalein being used as indicator. The more carbon the molecule of the compound contains, the greater the accuracy of the results. With alipathic hydrosulphides the end reaction is not sharp, and thioglycollic acid cannot be titrated with alkali either in aqueous or alcoholic solution. C. A. M.

On Quantitative Determination by Means of the Immersion Refractometer. H. Matthes. (*Chem. Ztg.*, 1906, xxx., 101.)—The author severely criticises some applications of the use of the immersion refractometer made by Wagner (*Ztschr. öffentl. Chem.*, 1905, xi., 404), especially in the case of sugar analysis. For legitimate uses of the instrument, such as the control of beer, wine, etc., prepared in the same way and under the same conditions, and the control of the strength of standard solutions, he refers to his own work (*Ztschr. analyt. Chem.*, 1904, xliii., 83). A. G. L.

Simplified Ultimate Organic Analysis for Scientific Purposes. M. Dennstedt. (*Chem. Zeit. Rep.*, 1905, xxix., 390.)—In the use of the simplified method (see ANALYST, xxx., 135) of the author a difficulty arises from the fact that, as some substances require different treatment in the combustion, these must be burnt in succession. The author now suggests some improvements.

The chief point of the "quick" method is that the boat containing the substance is not placed directly in the combustion tube, but in a narrow, hard glass tube, which may take any of three forms: (1) The form open at both ends, *a*, Fig. 1; (2) that with the hinder end closed, *b*, Fig. 1; or (3) that with a double current of oxygen, Fig. 2.

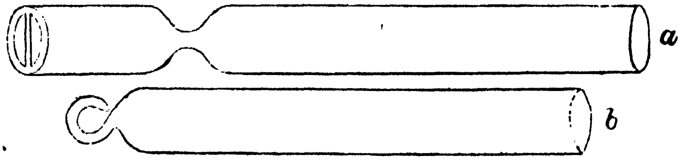


FIG. 1.

The last named is fitted with a capillary and a bubble counting arrangement through which the oxygen enters the inside of the tube. A T-piece of somewhat wider bore fits over the capillary, and is provided with a cork to fit into the main combustion tube. The main current of oxygen passes through the calcium chloride tube and the side tube of the T-piece. The bubbling apparatus and the calcium chloride tube are each connected to the ordinary drying apparatus by rubber tubing, which allows the tube to be withdrawn with one hand for introduction of the boat, etc.

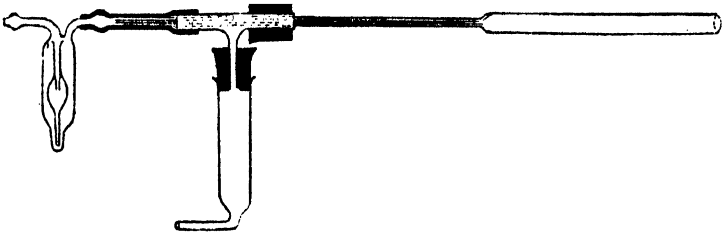


FIG. 2.

Form *a* is suitable for the combustion of substances which decompose slowly, with formation of much carbon difficult to burn—*e.g.*, sugar, albumin; form *b* is for use with easily volatile or moderately volatile substances, which do not give a separation of difficultly combustible carbon—*e.g.*, alcohols, benzol, aniline, etc.

The third kind of tube is serviceable for all substances without exception. The regulation of the combustion is especially easy, as this can be effected not only by the flame, but also by increasing or diminishing the rate of the inner current of oxygen.

The tubes can be obtained from Emil Dittmar and Vierth, Hamburg.

E. K. H.

INORGANIC ANALYSIS.

The Estimation of Opalescent Silver Chloride Precipitates. Roger Clark Wells. (*Amer. Chem. Journ.*, 1906, vol. 35, 99.)—A number of small precautions are described, which must be used in connection with the nephelometer (ANALYST, 1905, xxix., 203). It is found that ground-glass standards are to be preferred, and that for every concentration a suitable solution and excess of precipitant are required. It is also shown that the presence of electrolytes both augments the maximum opalescence and hastens its disappearance through coagulation. The best procedures in a number of special cases are also described. A. G. L.

The Volumetric Determination of Copper. G. Denigès. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 10-12.)—Ammoniacal solutions of cupric salts form double colourless compounds with alkali cyanides, thus:

$$CuSO_4 + 4NH_4CN = (NH_4)_2SO_4 + Cu(CN)_2.2(NH)_4CN.$$

The double cyanide thus formed is very stable, but gives up a portion of its cyanogen on treatment with silver nitrate, the amount varying, *e.g.*, with the degree of alkalinity of the liquid. The author has found that when the quantity of the copper salts taken contains from 1 to 50 mgms. of the metal, whilst 20 c.c. of $\frac{N}{10}$ potassium cyanide solution and 2·6 grams of ammonia are used, the total amount of liquid being 120 c.c., each cgm. of copper gives up to the silver an amount of cyanogen equal to 2·5 c.c. of the potassium cyanide solution, or in other words each c.c. of cyanide solution is equivalent to 4 mgms. of copper. In making a determination, a quantity v of the solution of the cupric salt containing not more than 50 mgms. of copper is treated with a volume v' of ammonia solution containing 2·6 grams of ammonia, and with 20 c.c. of $\frac{N}{10}$ potassium cyanide solution (standardized on the silver nitrate solution). Then, after the addition of 1 c.c. of a 10 per cent. solution of potassium iodide, and $99-(v+v')$ c.c. of water, the liquid is titrated with $\frac{N}{10}$ silver nitrate until there is a faint but persistent turbidity. If n c.c. be used, the value $(20-n)$ c.c. represents the cyanide taken up by the silver, and $(20-n)\times 0{\cdot}004$ gram gives the amount of metallic copper in the amount, v, of solution taken. In order to obtain concordant results it is necessary that the volume of $v + v'$ should not exceed 100. In testing cupric sulphate by this method the presence of zinc does not interfere with the results unless it amounts to 40 to 50 per cent. of the quantity of the copper salt.

Volumetric Determination of Copper and Mercury in Admixture with Each Other.—A quantity v (not exceeding 100 c.c.) of the solution, which should not contain more than 0·5 gram of mercury and 0·1 gram of copper, is treated with 5 c.c. of hydrochloric acid (specific gravity 1·18), and 5 c.c. of a solution (1 : 5) of cystallized sodium phosphite, and boiled for five minutes with continual agitation, after which it is filtered, and the filtrate and wastings made up to 150 c.c. The copper is then determined in 75 c.c. of this liquid in the manner described above, with the difference that it is necessary to add a constant of 0·8 c.c. to the volume of silver nitrate, n, consumed, to make up for the modification in the reaction caused by the added salts. The precipitate (containing the mercury) left on the filter is detached with the aid of a fine jet, and treated with 5 c.c. of water, 5 c.c. of hydrochloric acid, and 0·25 gram of potassium chlorate, the whole being heated until solution is complete, and the liquid then cooled and made up to 100 c.c. An aliquot portion (a c.c.) of the solution is now mixed with 12 c.c. of ammonia solution (25° Bé., S.G. 0·9), 10 c.c. of $\frac{N}{10}$ potassium cyanide solution, $100-n$ c.c. of water, and 1 c.c. of a 10 per cent. solution of potassium iodide, and titrated with $\frac{N}{10}$ silver nitrate solution. If q c.c. be used, $(10-q)=n$ represents the amount of cyanogen in terms of $\frac{N}{10}$ potassium cyanide solution given up by the mercury to the silver. This figure must be corrected by multiplying by the factor 0·96 if it lies between 0 and 5·5, or by the factor 1·04 and subtraction of 0·45 of the product if it lies between 5·5 and 9·5. The corrected value, c, multiplied by 0·02 gram (one ten-thousandth of the atomic weight of mercury) gives the quantity of that metal in the amount of solution, a, taken. C. A. M.

The Separation of Metals of the Arsenic Group. O. Materne. (*Bull. Soc. Chim. Belg.*, 1905, xix., 241.)—The mixed sulphides of arsenic, antimony, and tin are boiled with a 2 per cent. solution of borax containing some ammonium chloride (amount not stated), and the liquid allowed to cool and filtered. The filtrate contains the arsenic, which is precipitated by means of tartaric acid. The residue of the two other sulphides is treated with a boiling 5 per cent. solution of sodium carbonate (containing some ammonium chloride), which dissolves the antimony sulphide and leaves the stannous sulphide. On treating the filtrate with tartaric acid the antimony sulphide is re-precipitated, whilst the residue of stannous sulphide can be brought into solution by means of a 10 per cent. solution of sodium hydroxide.

C. A. M.

Determination of Carbon in Steel by Direct Ignition with Red Lead. Charles Morris Johnson. (*Proc. Eng. Soc. West. Pennsylvania*, 1906, xxi., 586.)—The author has found that for molybdenum and chromium steels the ordinary potassium cupric chloride method of determining carbon gives very discordant results, as the carbide obtained appears to oxidize rapidly if exposed to air, or, in some cases at least, to acid. He therefore burns such steels directly with red lead in a current of oxygen, the resulting carbon dioxide being absorbed in barium hydrate, and the barium carbonate formed filtered off and weighed. The baryta is contained in two large test-tubes, through which the carbon dioxide bubbles. The steel used should pass a 20-mesh sieve and be retained by a 30-mesh, or else it may be in the form of fine drillings. For high carbon steels, 0·5 to 1 gram is taken; for low carbon steels, 2 or 3 grams may be used. The sample is placed in a weighing-bottle with 4 grams of red lead, and well mixed by shaking; the mixture is then transferred to a porcelain boat and covered over with asbestos, which prevents creeping. The highest heat obtainable should be used for the combustion. Old boats may be cleaned with nitric acid. A combustion by this method takes in all only seventy-five minutes from start to reporting, and results obtained on ordinary steels check exceedingly well with those given by the usual method. A. G. L.

Determination of Sulphur in Roasted Zinciferous Ores and Similar Materials. G. Lunge and R. Stierlin. (*Zeits. angew. Chem.*, 1906, xix., 21.)—The authors show that the Watson-Lunge method is not applicable to materials containing zinc, and recommend the following: Exactly 2 grams of sodium bicarbonate, the alkali content of which is accurately determined, is mixed in a small nickel crucible with 3·206 grams of the finely powdered sample and 2 grams of potassium chlorate. The crucible is covered and heated for one hour, for the greater part of the time very gently, but finally to dull redness, without, however, allowing the mixture to fuse. The mixture is then extracted with water to which 25 c.c. of sodium chloride solution, absolutely neutral and free from magnesium salts, have been added. The whole is boiled until sodium chloride commences to separate out, when the insoluble residue is filtered off and washed with sodium chloride solution (to prevent iron oxide from passing through the filter). The filtrate is then titrated with hydrochloric acid, using methyl orange as indicator. The difference between the alkali value of the sodium bicarbonate taken and that found corresponds to the sulphur. If more than 6 per cent. of sulphur is present, only 1·603 grams of substance should be taken, and 2 grams of ferric oxide added, to prevent fusion of the mixture. A. G. L.

Titanium Chloride in Volumetric Analysis. E. Knecht and E. Hibbert. (*Chem. Zeit. Rep.*, 1905, xxix., 380.)—The authors have successfully used the strong reducing action of titanium chloride for the volumetric analysis of other substances, especially coloured organic compounds which form colourless "leuco-" bodies. This is usually effected by the addition of 2 atoms of hydrogen to the molecule. The colouring matter acts as its own indicator in the titration, as the end of the reaction is marked by the solution becoming colourless. The experiments must be conducted in absence of air, owing to the ease with which the "leuco-" bodies are oxidized. The authors also succeeded in accurately estimating inorganic substances by the same reagent. E. K. H.

Analysis of Incandescence Mantles. T. B. Stillman. (*Chem. Zeit.*, 1906, xxx., 60.)—The following scheme of procedure is given: Five mantles are burnt off, broken into small pieces, weighed, and evaporated with sulphuric acid in a platinum basin to dryness. This digestion is repeated four times. When cold, the mass is extracted with cold water, and the solution filtered, if necessary. The residue, if any, is fused with potassium hydrogen sulphate, diluted with water, and added to the main solution. Ammonium sulphide is now added in slight excess, and the precipitate collected on a filter and washed with water containing ammonium sulphide. The filtrate contains the calcium and magnesium, which are separated in the usual manner, after boiling the solution to drive off hydrogen sulphide, and filtering to remove separated sulphur. The precipitate contains the hydroxides of cerium, thorium, lanthanum, zirconium, aluminium, didymium, and yttrium. It is dissolved in hydrochloric acid, diluted with water, boiled; a dilute solution of sodium thiosulphate is added, the whole boiled for one hour, then allowed to stand for twelve hours and filtered, giving precipitate "A" and filtrate "B."

Precipitate "A" contains the oxides of thorium, zirconium, and aluminium. These are dissolved in hydrochloric acid filtered from precipitated sulphates; the latter are dried, ignited, fused with potassium hydrogen sulphate; the melt dissolved in cold water, precipitated with ammonia, filtered, and the precipitate dissolved in hydrochloric acid and added to the main hydrochloric acid solution. The latter is treated with an excess of sodium thiosulphate, boiled, filtered, treated with ammonia in slight excess, again filtered, and washed with water. The precipitate is dissolved in hydrochloric acid, supersaturated with oxalic acid, boiled for five minutes, allowed to stand for twelve hours, and filtered.

<table>
<tr><td rowspan="2">Precipitate. Wash with cold water, dry, ignite, and weigh as ThO_2. Gives no absorption spectrum.</td><td colspan="2">Filtrate. Treat with excess of sodium hydroxide, boil for ten minutes, filter, and wash with hot water.</td></tr>
<tr><td>Precipitate. Dry, ignite, and weigh as ZrO_2. Gives no absorption spectrum.</td><td>Filtrate. Acidify with hydrochloric acid, render feebly alkaline with ammonia; boil, filter, wash, and weigh as Al_2O_3. No absorption spectrum.</td></tr>
<tr><td>ThO_2.</td><td>ZrO_2.</td><td>Al_2O_3.</td></tr>
</table>

Filtrate "B" contains the oxides of cerium, yttrium, lanthanum, didymium, and thorium. The solution is rendered alkaline with ammonia, filtered, washed with cold water, the precipitate dissolved in hydrochloric acid, and the solution treated with dilute sodium thiosulphate solution to precipitate any thoria present. Any precipitate obtained is dissolved in hydrochloric acid and added to the hydrochloric acid solution of precipitate " A." The filtrate is precipitated with ammonia, filtered, washed, the precipitate dissolved in sulphuric acid; the excess of the latter is ignited, and the residue dissolved in cold water. The solution is now treated with a saturated solution of potassium sulphate, and the precipitate collected on a filter after standing for four hours, and washed with a solution of potassium sulphate.

<table>
<tr><td colspan="3">Precipitate. Dissolve in dilute hydrochloric acid, add oxalic acid, filter, dry, and ignite. Dissolve the residue in hydrochloric acid, precipitate with sodium hydroxide, saturate with chlorine gas, filter, and wash well.</td><td rowspan="3">Filtrate. Add a dilute solution of sodium hydroxide; filter off the precipitate, wash, dry, and weigh as Y_2O_3. Gives no absorption spectrum.</td></tr>
<tr><td rowspan="2">Precipitate. Dry, ignite, and weigh as CeO_2. Gives no absorption spectrum.</td><td colspan="2">Filtrate. Acidify with hydrochloric acid, boil, precipitate with oxalic acid, filter, wash, dry, and ignite. The residue is dissolved in nitric acid, and divided into two parts:</td></tr>
<tr><td>Test for Di with the spectroscope. A dark band shows that Di is present.</td><td>Add ammonium acetate, then ammonia, and filter. Treat the precipitate with solid iodine. Lathanum gives a blue colour, but no spectrum.</td></tr>
<tr><td>CeO_2.</td><td>Di_2O_4.</td><td>La_2O_3.</td><td>Y_2O_3.</td></tr>
</table>

The didymium may consist of praseodymium and neodymium; the salts of the former are green and of the latter rose-red. W. P. S.

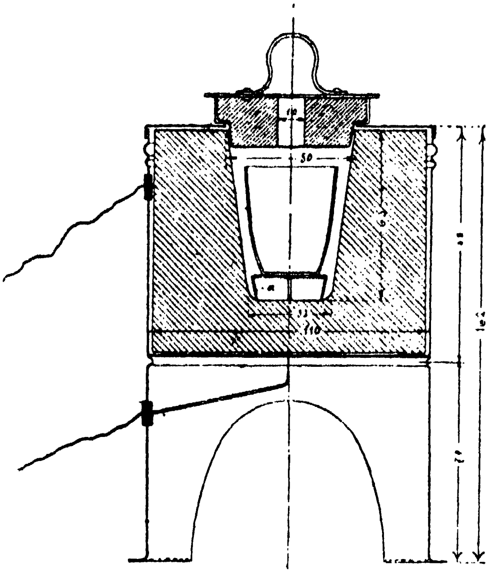
The Separation of Iodine from Chlorine and Bromine by Means of Hydrogen Peroxide. J. Jannasch and F. Zimmermann.

(*Berichte*, 1906, xxxix., 196, 197.)—The aqueous solution of the mixed halogens (120 to 150 c.c.) is treated with 15 c.c. of glacial acetic acid, and 3 c.c. of hydrogen peroxide (30 per cent.), which effects a quantitive separation of the iodine from the chlorine and bromine. The iodine thus liberated is distilled by means of a current of steam into three connected vessels containing respectively (*a*) 10 grams of hydrazine sulphate, and 10 c.c. of ammonium hydroxide in 80 to 100 c.c. of water, and (*b*) and (*c*) 0·5 gram of hydrazine sulphate, and 2 c.c. of ammonium hydroxide in 25 c.c. of water. After the whole of the iodine has passed over, the contents of the absorption vessels are cooled, mixed, and acidified with 30 to 40 c.c. of strong sulphuric acid, and the iodine determined as silver iodide. C. A. M.

The Detection and Determination of Chlorate in Sodium Nitrate. L. Grimbert.

(Journ. Pharm. Chim., 1906, xxiii., 98-100.)—A few c.c. of a solution of the sodium nitrate are shaken with one drop of aniline, and a little strong sulphuric acid then poured down the side of the tube. In the presence of a chlorate an intense blue zone appears at the junction of the liquids. For the determination of the chlorate a given weight of the sodium nitrate is ignited with a little cane sugar to effect reduction, the mass extracted with water, and the chloride determined by titration in the usual manner. In this way the author found a sample of sodium nitrate to contain 0·692 per cent. of sodium chlorate. Three other samples of sodium nitrate free from chloride also contained chlorate, but different samples of potassium nitrate examined were found to be quite pure. C. A. M.

Use of the Electric Furnace for Burning Magnesium Phosphate Precipitates. F. Haussding.



(Chem. Zeit., 1906, xxx., 60, 61.)—The construction of the furnace is shown in the illustration. The crucible containing the precipitate of ammonium magnesium phosphate rests on a quartz-glass triangle or on a piece of platinum foil placed on the bottom of the furnace. The lid of the furnace is also covered with platinum foil. After passing a current of 1·5 ampères at a pressure of 220 volts through the furnace for twenty minutes, it is ready for use. Only two minutes' heating are required to obtain a completely white residue of magnesium pyrophosphate, and there is no danger of the high temperature causing loss of phosphoric acid. The use of the furnace does away with the injurious action of ordinary gas-flames on the platinum crucibles. W. P. S.

The Determination of Carbon Monoxide in Air. A. Levy and A. Pécoul.

Comptes Rendus, 1906, cxlii., 162.)—The method is based upon the fact, recorded by Gautier, that carbon monoxide, even when only present in traces in air, reduces iodic anhydride, and also on the colorimetric determination in a chloroform solution of the iodine thus liberated. Acetylene has a similar reducing action, but to a much smaller extent. Thus, air containing 4 parts per 10,000 of acetylene causes only a very slight reduction of the iodic anhydride, whereas with 1 part of carbon monoxide in 10,000 a large amount of iodine is liberated. Hence in testing ordinary air for carbon monoxide no special precautions are necessary, but in the case of industrial gas-products any acetylene, hydrogen sulphide, etc., must be absorbed before making a determination. The most accurate results are obtained by using a solution of potassium hydroxide for the absorption of the iodine. C. A. M.

Determination of Water in Superphosphates and in Silicofluorides. Ludwig Schucht. (*Zeits. angew. Chem.*, 1906, xix., 183.)—To determine *moisture*, as distinct from chemically combined water, in *superphosphate*, 2 grams of the sample are rubbed up in a glass dish with 20 c.c. of absolute alcohol. After one hour the liquid is decanted through a weighed filter-paper; the insoluble material is brought on to the filter with more alcohol, washed with ether, dried at 40° C., and weighed. The filtrate is evaporated to dryness, and the residue dried at 120° C. to constant weight. The weighings must be made as quickly as possible. The difference between the original 2 grams and the sum of the two residues gives the moisture. For the determination of *free acid*, the author recommends the use of his oxalate method, and answers some objections which have been brought against it.

To determine the free acid in commercial *silico-fluoric acid*, 50 c.c. are weighed out, diluted with water to 1 litre, and 50 c.c. of this solution titrated hot with $\frac{N}{2}$ sodium hydroxide, using phenolphthalein as indicator. If free hydrofluoric acid is also present, the total acid is determined in this way, and the precipitate produced by potassium chloride and ethyl alcohol by Stolba's method is also weighed. The precipitate may also be titrated with sodium hydroxide.

In the analysis of commercial *sodium silico-fluoride*, *moisture* is determined by heating 2 grams of the substance for three hours at 100° C. in a current of dry air which has to bubble through $\frac{N}{2}$ sodium hydroxide after passing over the substance, any volatilized acid being thus retained and allowed for. To determine *free acid*, 3 grams of the sample are moistened with methyl orange in a glass dish, and titrated with $\frac{N}{2}$ sodium hydroxide. To determine the sodium silico-fluoride, 3 grams of the sample are dissolved in 450 c.c. of water, and the solution is titrated hot with $\frac{N}{2}$ sodium hydroxide, using phenolphthalein as indicator. The free acid previously found must be deducted from the result, and the proportion of sample to water should always be that stated, otherwise errors due to hydrolysis will be made. A. G. L.

Determination of Available Plant Food in Soil by Use of Weak Acid Solvents. A. D. Hall and A. Amos. (*Proc. Chem. Soc.*, 1906, xxii., 11.)—Repeated extraction, with water charged with carbon dioxide or with a 1 per cent. solution of citric acid, of soils of known history has shown that the first extraction does not remove the whole of the soluble phosphates, the reaction being a reversible one. With carbon dioxide and water the position of equilibrium is approximately constant for successive extractions, but with dilute acetic acid the amount of phosphoric acid dissolved falls for the first four or five extractions, then becoming nearly constant. No support is found for the theory that all soils establish in the soil-water a solution of phosphoric acid of approximately the same composition, and independent of the fertilizers the soil receives. W. H. S.

Mechanical Analysis of Soils. J. A. Murray. (*Chem. News*, 1906, vol. 93, p. 40.)—The author has worked out the following scheme for the mechanical analysis of soils : Five grams of the air-dried fine soil (passed through a 100-mesh sieve) were disintegrated in weak ammonia solution and allowed to stand for fifteen minutes, after which the water containing the finer material in suspension was poured off. The coarser material was then introduced into a 200 c.c. Erlenmeyer flask, which was filled with water and attached by a rubber joint to a wide glass-tube of exactly the same diameter as the neck of the flask, from which the flange had previously been removed. The tube used was 147 cm. long and 2·3 cm. internal diameter. Water was next poured gently down the side of the tube so as to fill it, after which it was closed by a cork and inverted in a large glass basin full of water. The cork was then immediately removed and a small weighed porcelain dish placed under the open end of the tube under the water. The particles descending the tube were collected in a series of fractions, the first porcelain dish being removed and another one substituted five minutes after the first particle had reached the bottom, the second dish being removed after another ten minutes, and the third after another twenty minutes (or else, after another twenty-five minutes, the times as given not corresponding exactly with each other). Nearly the whole of the remainder of the material was deposited in another hour. This last fraction was returned to the beaker containing the quantity of fine material originally poured off, well mixed with it, and the whole then placed in the flask and allowed to fall through the tube as before, the same water being used so as to avoid loss of material. In this way three more fractions were collected, after one, three, and seven hours respectively. The liquid remaining in the flask and tube was then measured and an aliquot part evaporated to dryness so as to give a seventh and last fraction of finest material. The other fractions collected were also dried and weighed. The preliminary separation into finer and coarser material is necessary to prevent some of the fine particles being dragged down with the coarse material. The method enables a soil to be separated into a number of fractions containing particles of nearly uniform size; duplicate analyses made by it are in good agreement with each other.

A. G. L.

A Rapid Method for the Determination of Moisture in Fuels, especially Coal. E. Graefe. (*Braunkohle*, 1906, iv., 581; through *Chem. Zeit. Rep*,. 1906, xxx., 23.)—Ten grams of the coal are heated with 50 c.c. of solar oil or petroleum in a 75 c.c. distilling flask until 25 c.c. of oil have been distilled off, which should require about five minutes. The distillate is caught in a 25 c.c. measuring cylinder, the amount of condensed water being read off directly; each $\frac{1}{10}$ c.c. corresponds to 1 per cent. of moisture in the coal. The neck of the distilling flask should only reach a short distance above the side-tube, and a narrow condenser tube (above 5 mm. diameter) should be used. The method is stated to give serviceable results.

A. G. L.

Standardization of Chemical Disinfectants. (*Chem. Trade Journ.*, 1906, xxxviii., 4.)—The large variety of these now on the market renders desirable their classification according to their efficiency, and since they are sold for germicidal purposes, any comparison should be on this basis. The need for standardization is emphasized by the following table, taken from the *Public Health Engineer*.

Sample.	Carbolic Acid Coefficient.	Price per Gallon, or 10·5 lbs.	Cost of Disinfectant equivalent to 1 Gallon of Carbolic Acid.
		£ s. d.	£ s. d.
A.	0·02	0 3 6	8 15 0
B.	0·30	0 5 6	0 18 4
C.	8·00	0 4 0	0 0 6
D.	1·00	0 1 0	0 1 0
E.	2·50	0 8 0	0 3 2
F.	1·40	0 2 6	0 1 9
G.	0·30	7 17 6	26 5 0
H.	0·10	0 18 9	9 7 6
I.	0·10	0 15 0	7 10 0
J.	0·90	0 7 6	0 8 4
K.	2·50	0 3 6	0 1 5
L.	0·03	2 0 0	66 13 4
M.	11·00	0 4 0	0 0 4

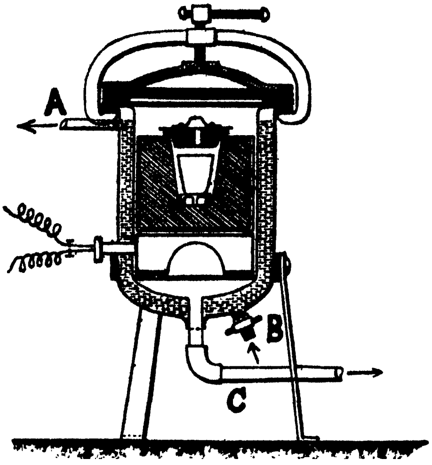
W. H. S.

Determination of Turbidity of Water. J. F. Liverseege. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 45.)—Test type as used by oculists, mounted on a board and attached to a cord running on a pulley, is viewed through a 2-foot tube, filled with the water under examination, and placed with its end about 6 inches from the type, which is raised or lowered till a set is found that can be easily read. The turbidity figure is obtained by subtracting 2·5 feet from the normal range in feet for the particular type, and is recorded as "turbidity expressed in feet of distance required to read standard type." If a water is so turbid that 2 feet of it obscures the type entirely, a 1-foot tube may be used, still reading at 2·5 feet from the type, but the results cannot be calculated from one tube to the other. W. H. S.

Filtration of Finely Divided Precipitates. C. S. Palmer. (*Chem. Zeit. Rep.*, 1905, xxix., 342.)—The author proposes to add 1 or 2 drops of albumen to the liquid to be filtered, and, after stirring, to quickly raise to boiling. The coagulated albumen enables the precipitate to be easily filtered, washed, and burnt. The trace of ash from the albumen is quite negligible. E. K. H.

APPARATUS.

A Form of Burette for avoiding Errors in Titration at the Boiling-point. H. Göckel. (*Chem. Zeit.*, 1905, xxix., 1208.)—A modification of Koninck's burette, in which the exit-tube comes out at right angles to the main tube. In the present type the side tube is not horizontal, but curves up somewhat and then down again. This avoids the necessity of placing the burette at a high level. The burette can be obtained from Dr. H. Göckel, Berlin, W. E. K. H.

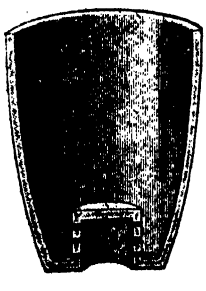


Heating under Reduced Pressure by the Aid of an Electric Oven. E. Haagn. (*Chem. Zeit.*, 1905, xxix., 1209.)—The accompanying figure shows a very useful form of electric oven, furnished with an arrangement for giving a partial vacuum. The bottom portion of the apparatus is made with a double wall, between which water circulates, being admitted by the aperture B, and running off by aperture A. In consequence of this cooling, the top can be safely provided with a rubber fitting, which insures an air-tight joint. The oven is exhausted by means of the tube C, connected to a water-pump.

The oven itself is a modification of that designed by W. C. Heræus, of Hanover, but, in contrast to the earlier models, the heating resistance is within the mass of the oven body. This gives greater heat economy in working, and causes the highest temperature to be more quickly attained. A temperature of 1100° C. can be obtained, and this is reached in about twenty to twenty-five minutes. About $\frac{1}{2}$ kilowatt is used.

For ordinary purposes a good water-pump gives a sufficiently reduced pressure; but, of course, any desired reduction can be obtained by the ordinary means. The advantages of heating in a partial vacuum are often very great. Thus marble, which requires a good half-hour in a gas furnace for complete conversion to lime, is, in the present apparatus, completely converted in three minutes after the full temperature is attained. By first exhausting and then admitting any gas, it is easy to use this apparatus for heating substances in an atmosphere of any desired gas.

The advantages of electric heating are also obtained by this oven. The influence of gas flames on platinum crucibles is especially bad, and the avoidance of this evil is almost a sufficient recommendation. E. K. H.



An Improved Pattern of Gooch Crucible. H. Vollers. (*Chem. Zeit.*, 1905, xxix., 1088.)—The principle of this form of crucible is clearly indicated in the accompanying figure. In place of the usual perforated bottom, there is a raised cylinder with the holes passing through the wall, the direction of the holes being parallel to the bottom.

By this device the author claims to have removed the defect shown by an ordinary Gooch crucible, that the pores get blocked up by the asbestos; the asbestos can be pressed down very firmly, and, if the filtration is then too slow, some of the holes, usually only a few, can be cleared by means of a bent needle.

The crucible is supplied by the firm of C. Gerhardt, Bonn a. Rh. E. K. H.

A Constant Temperature Bath for Low Temperatures. A. Given. (*Journ. Amer. Chem. Soc.*, 1905, xxvii., 1519.)—The bath shown in Fig. 1 was designed originally to keep pyknometers at a constant temperature of 15·6° C.; but it has since been found to work well at other moderately low temperatures. In principle it consists essentially of a bath of distilled water, in which the greater part of the pyknometer is immersed, and which is cooled by an ice-box, and heated by a Bunsen burner (not shown) fitted with a gas-regulator. The bath itself is double-walled, the space between the outer galvanized iron wall and the inner copper wall being packed with asbestos. The ice-box *I* is of copper, and fitted with a wooden cover. It is raised 1½ inches above the bath, so as to leave space for an overflow-pipe, and it extends to within 1½ inches from the bottom of the bath. A gentle current of air is introduced close to the bottom of the ice-box, and serves to stir the water in the outer bath. The pyknometers stand on the perforated shelf *P*, supported on hangers, which also carry the narrow shelf *S* for the thermometer and gas-regulator. This last is shown in Fig. 2, and consists of a large bulb, *A*, 5 inches long and 1 inch in diameter, filled with methyl alcohol (toluol might be even better). *M* represents the mercury seal, *R* being a rod of glass to strengthen the apparatus. The gas enters at *I* and leaves at *O*; at *P* there is a hole for the pilot-flame supply.

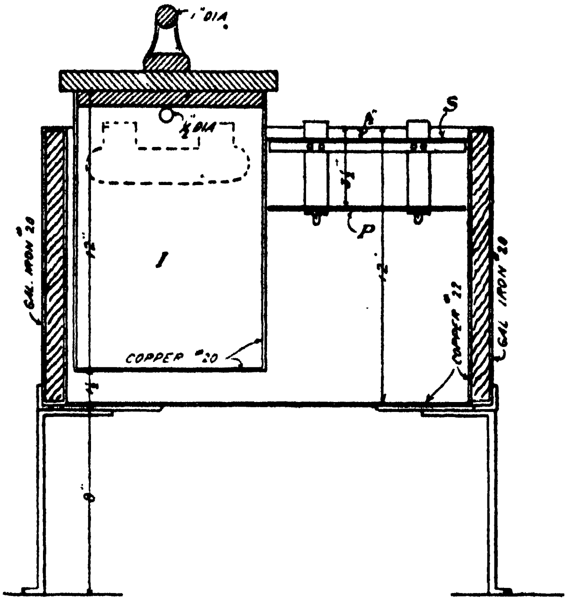


FIG. 1.

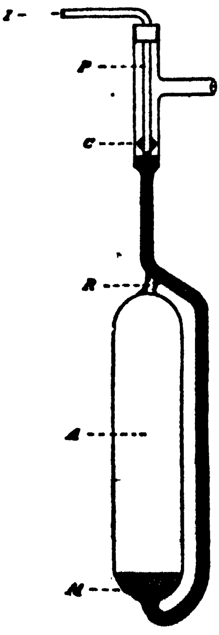
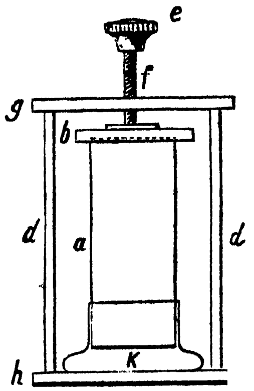


FIG. 2.

In using the bath, the pyknometers are first of all cooled to about 14° C., and then only placed in the bath, in which they may remain for any reasonable time. A bath, 18 inches square and 12 inches deep, consumes about 50 pounds of ice in seven hours in the hottest weather, but less than 1 pound of ice in it will keep the temperature of the bath constant.

A. G. L.

A Modification of Gintl's Pyknometer. M. Raukusin. (*Chem. Zeit.*, 1905, xxix., 1087.)—The author brings forward a modified form of apparatus, the design of which is seen in the accompanying figure.

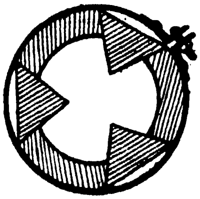


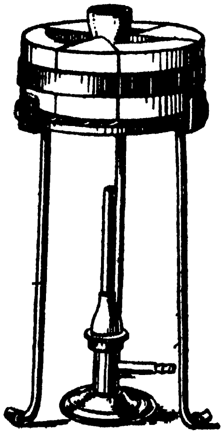
The sample of fat is contained in the cylinder *a*, which in this form is open at *both* ends. The lower end of this pyknometer tube is ground to fit the glass cap *k*, which is ground on the inside, and thus a tight joint closes the tube. On the upper edge of the pyknometer tube a glass cover, *b*, is laid, which closes the tube tightly through receiving the wall of the tube in a small annular canal.

The apparatus is used as follows: The three portions of the instrument are carefully cleaned and dried. The tube *a* is then placed in the cap *k*, and filled with the melted fat. The cover *b* is then laid on the tube, so that the wall of the tube fits exactly with the canal in the cover. The whole instrument is then placed in a metal clamp, shown in the figure, and the excess of fat removed by pressure from the screw *f*, and the instrument then washed with petroleum ether and cleaned with chamois leather.

The fat can be removed from the cylinder by a glass rod, the last traces being got rid of with ether, etc. E. K. H.

A New Crucible Triangle. A. Kette. (*Chem. Zeit.*, 1905, xxix., 1208.)—The author points out the failings of the ordinary pipeclay triangle and the platinum triangle. In particular all forms suffer from the defect that the flame is broken on the bottom of the crucible, and its further heating power is thus lost. To remedy this defect, the author brings forward the arrangement shown in the figure.





The crucible rests on the angles of the three prisms, and the flame touches only fire-resisting material. The ring surrounding these pieces can be tightened and loosened by means of the small screw, and the opening thus altered to fit various-sized crucibles. Finally, the effective heating of any given flame is much increased, as very little of its power is lost.

The author states that a platinum crucible can be easily and quickly raised to a temperature of 1300° C. This apparatus can be obtained from the firm of Bender and Hobein, Munich. E. K. H.

A New Vacuum Filter for Laboratory and Domestic Use and a wholly Novel Method of Cleaning the Filtering Material. (*Zeits. angew. Chem.*, 1906, xix., 95.)—In the vacuum filter described the filtering material may be easily cleaned by evacuating the upper part of the apparatus by means of the pump *p*, as shown in Fig. 1, air being sucked in through the opening F below the filter. As soon as the filter is thoroughly clean, the liquid in A will become filled with small air-bubbles. The air admitted through F may be previously sterilized in any convenient way. Fig. 2 shows the normal way of working the apparatus as a filter. It may be obtained of the following firms: Warmbrünn, Quilitz and Co., Berlin, N.W., Haidestrasse 55 to 57; Albert Dettloff, Berlin, N.W., Luisenstrasse 59; Franz Hugershoff, Leipzig.

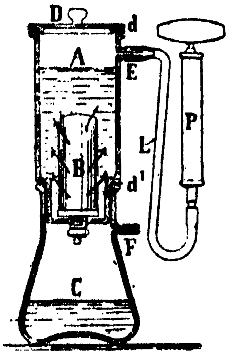


FIG. 1.

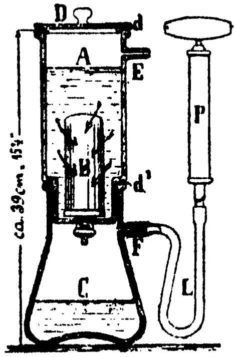
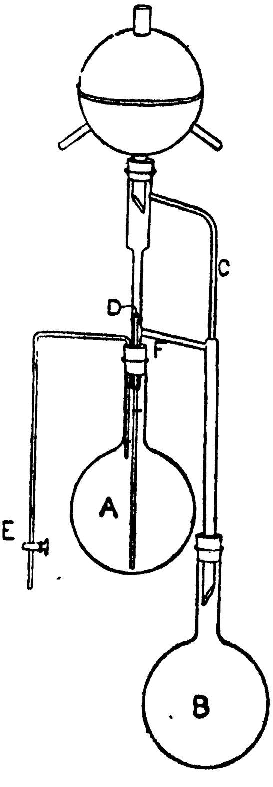


FIG. 2.

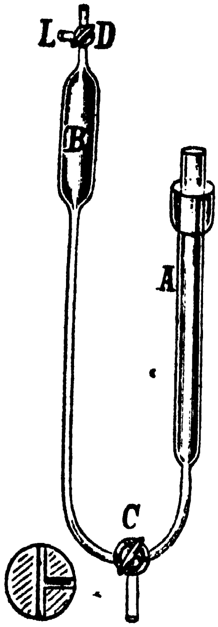
A. G. L.

Apparatus for Continuous Extraction of Liquids with Ether, Benzene, or other Solvent. R. S. Bowman. (*Proc. Chem. Soc.*, 1906, xxii., 24.)—The apparatus described obviated the use of numerous corks and rubber joints, and may be easily constructed in the laboratory.

The flask A, which may frequently be replaced by a wide test-tube, is filled almost to the neck with the liquid to be extracted, one-eighth to one-fifth of this volume of solvent being placed in the flask B, which is suitably heated. The vapour passes up the tube C, is condensed, and falling back, collects in the inner tube D, forming a column of liquid which in time becomes long enough to force its way through the liquid in A, and small drops of solvent, ascending in A, collect on the surface of the liquid, and finally overflowing at F, return to the flask B. On completion of the extraction, the solvent is run off from the neck of the flask by opening the stop-cock E.



W. H. S.

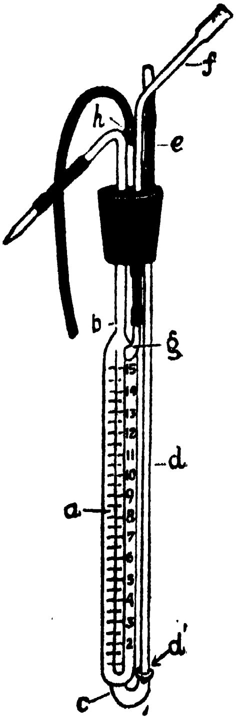


Apparatus for Removing Gases from Aerated Liquids before Determining the Specific Gravity of the Latter. K. Ulrich. (*Chem. Zeit.*, 1906, xxx., 90.)—The liquid is filled into the cylinder A and drawn into the bulb B by attaching the pipe L to an air-pump, the taps C and D being turned to give the necessary connections. The tap C is then closed, and the action of the pump allowed to proceed until the gases have been completely removed from the liquid in B. Air is now admitted by turning the tap D, and the liquid allowed to flow back again through the tap C into the cylinder A, where its specific gravity is taken by means of a hydrometer. The short tube below the tap C serves for emptying the apparatus.

W. P. S.

Note on a Combined Wash-Bottle and Pipette. J. W. Hogarth. (*Chem. News*, 1906, vol. 93, p. 71.)—By inserting the apparatus shown in an ordinary wash-bottle flask, a measured volume of solution may be delivered from the jet of the wash-bottle. The measuring vessel *a*, which is fitted with a jet and the mouth-piece *f*, is filled by raising the rod *d*, which closes the lower orifice, *d'*, of *a*, and by blowing into the wash-bottle flask through the rubber-tube *h*. As soon as *a* is filled to the desired extent, the rod *d* is released, when the rubber-tube *e*, which connects *d* and the short piece of glass-tubing in the stopper through which it passes, forces the rod back into position, so as to close the lower opening of *a*. The vessel *a* is capable of holding 15 c.c., and of delivering any less quantity with an error of only $\frac{1}{5}$ c.c.

A. G. L.



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MAY, 1906. Vol. XXXI., No. 362.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, April 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. E. Quant, F. E. Thompson, and F. W. M. Ross, were read for the second time; and certificates in favour of Messrs. Henry George Harrison, M.A. (Cantab.), A.I.C., 1, Park Road, New Cross, S.E., assistant to Dr. Rideal; Edward Miller, 81, Chardmore Road, Stoke Newington, N., assistant chemist to the Aylesbury Dairy Company, Limited; and Francis William Passmore, Ph.D. (Würzburg), 63, Queen Victoria Street, E.C., analytical and consulting chemist, were read for the first time.

Messrs. G. Craig, J. B. Gall, H. N. Hanson, B. W. Methley, F. D. Ratcliff, and F. Robertson, were elected members of the Society.

The following papers were read: "The Influence of Organic Matter on the Efficacy of Certain Disinfectants," by M. Wynter Blyth, B.A., B.Sc.; "The Determination of Higher Alcohols in Spirits" (II.), by Philip Schidrowitz, Ph.D., and Frederic Kaye, A.R.C.Sc.; and "A New Method of Estimating Moisture, with Special Application to Moisture in Cordite," by P. V. Dupré.

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ANNUAL ADDRESS OF THE PRESIDENT.

(*Delivered February* 7, 1906.)

WHEN, a year ago, the Society did me the honour of electing me President, I undertook the duties with considerable trepidation. I desire at the outset of my address to say that if my year of office has been attended by any small measure of success, it is due to the very kind way in which my fellow-officers, the Council, and the members generally, have co-operated. I take this opportunity of thanking them all for their assistance and forbearance.

In the past year we have unfortunately lost two members, Messrs. Ackroyd and Tichborne, by death, and eight by resignation.

On the other hand, we have an increase in membership of nineteen. The total membership is now 310.

During the visit of certain members of the American Section of the Society of

Chemical Industry, it occurred to some of us that it would be a graceful act, and one which would do honour to the Society, to elect as Hon. Member Dr. Wiley, Chief of the Bureau of Chemistry of the United States. Dr. Wiley's reputation is known to us all.

The Society has held eight meetings, at which twenty-four papers were read and discussed. The following is a list of the papers:

"The Estimation of Oxygen in Copper." By S. Dickson.

"Some Conditions affecting the Ether Values of Brandies." By P. Schidrowitz, Ph.D., and F. Kaye, A.R.C.Sc.

"The Determination of Higher Alcohols in Spirits." By P. Schidrowitz and F. Kaye.

"The Action of Slightly Alkaline Waters on Iron." By Cecil H. Cribb, B.Sc., and F. W. F. Arnaud.

"The Separation of Strychnine and Brucine." By D. Lloyd Howard.

"Ammonium Oxalate: Its Formula and Stability." By P. V. Dupré.

"Notes on some Abnormal Milks from Cleveland and South-East Durham." By A. C. Wilson.

"A Simple and Convenient Camera for Photomicrographic Work." By A. C. Wilson.

"The Composition and Analysis of Milk." By H. Droop Richmond.

"Note on an Objectionable Method of Fining Wine." By R. Bodmer.

"Note on the History of Distilled Spirits, especially Whisky and Brandy." By T. Fairley.

"A Rapid Method for the Determination of Tin in Copper-Tin Alloys," and "Water from the Simplon Tunnel." A. G. Levy.

"Notes on 'Dilka,' or Surin Fat." By J. Lewkowitsch.

"The Determination of Oxygen in Copper." By L. Archbutt.

"The Reducing Action of Hydrogen," "The Estimation of Traces of Arsenic by the Marsh-Berzelius Method," and "Insensitiveness of Zinc." By A. C. Chapman and H. Law.

"Note on the Removal of Arsenic from Hydrochloric Acid for Use in the Marsh-Berzelius Method." By A. R. Ling and T. Rendle.

"Discussion on Brandy." By Otto Hehner.

"The Volumetric Estimation of Reducing Sugars." By A. R. Ling and T. Rendle.

"The Inversion of Cane Sugar in the Presence of Milk Constituents." By the Hon. Francis Watts.

"The Colorometric Estimation of Salicylic Acid in Food-stuffs." By F. T. Harry and W. R. Mummery.

"Notes on Preservatives." By Edwy Godwin Clayton.

Many of these papers were of very great value, and it would be invidious to mention any one specially.

Most, if not all of them, have been published in the ANALYST, which, under the able editorship of our friend Dr. Sykes and the supervision of the Publication Committee, continues to hold the high position it has hitherto attained.

The financial position of the Society is not quite so satisfactory as one could desire, though there is no need for alarm. In the hands of our able Hon. Treasurer, Mr. E. W. Voelcker, who will explain in detail the state of affairs, we may, I think, feel secure.

But I should like to take this opportunity of saying that further increase of membership is desirable, since the journal of the Society necessarily makes considerable demands on our income.

There are many friends whom we should be glad to welcome as members of the Society, and I would urge upon you the importance of endeavouring to induce any such to join. The scope of the Society is wide enough even at present to embrace those who are not necessarily Public Analysts, but who, according to the constitution and rules, are "engaged in the profession of analytical chemistry."

I shall refer again to this matter when speaking of the proposed incorporation of the Society.

The Council, and the committees appointed by it, have been engaged on various matters of interest to the members generally.

Thanks largely to the initiation and activity of our senior Hon. Secretary, Mr. A. C. Chapman, a scheme has been promulgated for the purpose of fostering research in analytical problems.

A list of subjects has been prepared and submitted to several professors of chemistry in the various colleges. It is satisfactory to know that the scheme has not only been warmly approved, but that a number of researches are now actively in progress.

I may mention that three of the papers brought before the Society during the last year—viz., "The Estimation of Salicylic Acid in Food-stuffs," by Messrs. Hardy and Mummery; "The Determination of the Higher Alcohols in Spirits," by Dr. Schidrowitz; and "The Separation of Strychnine and Brucine," by David Lloyd Howard—are the outcome of this scheme.

Acting upon a suggestion made some time ago by Mr. Carteighe on behalf of the Pharmaceutical Society, which received the hearty approval of our Council, a joint committee—or, as we propose to call it, an Advisory Committee—consisting of three representatives of each Society, has been formed. Their opinion and advice will be at the disposal of members of the Society in cases where difficulties may be met with as to the true composition of drugs. Cases undoubtedly have arisen in the past where a reference to such a body would most probably have resulted in preventing trivial or even vexatious prosecutions. When I tell you that the Committee consists, among others, of Sir Thomas Stevenson, Mr. Carteighe, Mr. John Umney, and Mr. Edmund White, I think you will agree with me that its deliberations are certain to be judicial. Details of the scheme will be issued to members in due course.

Some months ago the Board of Agriculture issued a circular to local authorities, the purport of which was to induce them to refrain from instituting proceedings against vendors of milks which proved on analysis to be below the standard, if there was reason to believe that such deficiencies were not fraudulently brought about.

I have no hesitation in saying that the effect of such a circular is most pernicious. The Board of Agriculture adopted the standards recommended by the Departmental

Committee, which sat for many days and heard masses of evidence, and any modification of these standards can only, in my opinion, be to the advantage of dishonest men.

That harm has come of this circular is shown by the following pronouncement by Mr. d'Eyncourt, stipendiary magistrate: " Mr. d'Eyncourt said that his reason for refusing the summons was that analyses were all a question of inference, and a margin of 3 per cent. was not sufficient. He was bound to say that if such a case were proved before him he should inflict no penalty, as samples of admittedly genuine milk had been known to contain 10 to 15 per cent. more water than the standard allowed. Therefore it would be simply wasting time to grant a summons."

Personally, I am of opinion, in view of Section 4 of the Sale of Food and Drugs Acts, 1899, and the fixing of the standards by the Board of Agriculture, no magistrate has the right to refuse to grant a summons. The summons should be issued and the case tried on its merits. This I have reason to believe is also the view of the officials of the Board of Agriculture. Section 4 says:

> "The Board of Agriculture may, after such inquiry as they deem necessary, make regulations for determining what deficiency in any of the normal constituents of genuine milk, cream, butter, or cheese, or what addition of extraneous matter or proportion of water in any sample of milk (including condensed milk), cream, butter, or cheese shall for the purposes of the Sale of Food and Drugs Acts raise a presumption, until the contrary is proved, that the milk, cream, butter, or cheese is not genuine or is injurious to health, and an analyst shall have regard to such regulations in certifying the result of an analysis under those Acts."

Years ago a Departmental Committee of the Local Government Board was appointed to consider the question of the use of preservatives in foods. After hearing a large amount of evidence, certain valuable recommendations were made. It is a matter of great regret that up to the present time no legal effect has been given to these recommendations, though provision was made in the Sale of Food and Drugs Acts for such procedure in Section 4—at any rate as regards milk, cream, butter, and cheese.

It has happened on at least two occasions that when the question of fees to be paid to Public Analysts was under consideration by local authorities, comparison was made between the fee of 10s. 6d. paid for analyses of milk and the 6d. or 1s. fee paid by farmers to certain agricultural colleges which are State-supported. Some time ago I drew the attention of Sir Thomas Elliott, the Secretary to the Board of Agriculture, to the matter, and he was good enough to send me the following letter, with permission to make any use of it I liked:

[Copy].

A 14318/1905.

Board of Agriculture and Fisheries,
4, Whitehall Place,
London, S.W.
December 2, 1905.

Sir,—I am directed by the Board of Agriculture and Fisheries to advert to your letter of the 16th ult., addressed to Sir Thomas Elliott, and to say that they are of opinion that the services rendered in return for the small fees charged by those Agricultural Colleges who have undertaken, on behalf of farmers, to make frequent tests of samples of milk cannot properly be regarded in the same light as those for which the ordinary fees for an exact and chemical analysis are charged by a Public Analyst.

The view of the Board is that there is an essential difference between the rapid mechanical tests carried out by the Agricultural Colleges, for the purpose of enabling farmers to effect an improvement in the economical management of their dairies, and the important and accurate analyses required of Public Analysts, with the object very often of furnishing evidence on which to base a prosecution under the Sale of Food and Drugs Acts.

In the first case, it should be understood that the tests are made at the request of the farmer, and for his private information only. The test applied is that known as the Gerber test, which merely calls for care and a certain amount of practical skill, but does not necessitate any previous scientific training. It is intended that the tests should be taken regularly and continuously, so that each test becomes one of a series which by comparison will enable the farmer—(1) to perceive the variations in the quality of his milk, as regards the contents of butter-fat; (2) to attribute such variations to the correct cause—*i.e.*, either to a change of weather, the length of time between the hours of milking, the food-supply, or the housing and treatment of the cows, etc.; and (3) to discover under what conditions the best supply of milk is obtained, which cows are the most profitable, and, generally speaking, how his business may be conducted to the best advantage.

For this purpose it is not necessary that the tests should be of the nature of an exact analysis; and so long as the result is sufficient to enable the farmer to determine his position, as above indicated, nothing more is required.

There can be, however, no question that analyses conducted by Public Analysts at the request or on behalf of local authorities must necessarily be carried out in the most scientific and skilful manner, in order to insure with the utmost degree of exactness the character of the material analysed. Where so much depends on the result of the analysis, it would not be possible to adopt a rapid mechanical test in the same manner as can be used with advantage for determining approximately the butter-fat constituents in samples of milk submitted by farmers.

The Board do not consider that the remuneration paid to Public Analysts on the usual scale recognised by the Society of Public Analysts is excessive, and they believe that if that remuneration were to be reduced to the level of the fees charged by Agricultural Colleges for making tests of farmers' milk, efficient scientific work could not be reasonably expected, and the public confidence in the efficacy of analytical checks on food adulteration would be considerably impaired.

I am, sir,
Your obedient servant,
(Signed) W. Somerville,
Assistant Secretary.

Not only was this letter sent, but further reference was made to the subject in the January number of the journal of the Board of Agriculture.

This action of the Board of Agriculture is especially interesting as evidence of the friendly feeling which exists towards Public Analysts.

As regards the effect of granting facilities to farmers to have their milk analysed, I am inclined to think that more harm than good will ensue. Properly fed and housed, cows will with rare exceptions give milk of standard quality. If farmers find that their milk contains 4 per cent. of fat, it will be an inducement to the dishonest to reduce it to 3 per cent. and sell butter at the same time.

It is more important than ever that the Public Analyst should be paid an adequate fee. Adulteration is daily becoming more scientific, and its detection more and more difficult. It is greatly to be feared that this scientific adulteration is largely due to the misuse of talent by some unscrupulous analysts, who thus prostitute an honourable profession. To take one instance: the adulteration of butter by the addition of cocoanut oil is so ingeniously done that one can only suppose it is due to the exercise of a skill that might be much better employed. Again, the admixture of water with butter to the extent of 30 per cent. or more under the guise of milk-blending is ingenious, but in this case the ingenuity of the chemist is more than equalled by that of the lawyers who are responsible for the framing of the saving notice, which up to the present has been successful in shielding the manufacturers.

A question of considerable interest to those members of the Society who are Public Analysts is that of the appointment of whole-time analysts. It is an open question whether or not such appointments are legal. It is evident that they are not contemplated by those responsible for the framing of the Sale of Food and Drugs Acts. Be that as it may, it is important that, if made, such remuneration should be offered as to attract analysts of experience and repute. The City Council of Bristol have lately advertised for an analyst, and the duties he is to be called upon to perform are so numerous and diverse that no one man can be expected to perform them adequately. The remuneration offered is even below that limit which some years ago a certain well-known statesman regarded as the maximum. As, however, it is probable that the statesman in question has altered his views on this subject owing to recent political changes, there is some hope that an appeal to his department on the question of fees will be sympathetically received. The Council of the Institute of Chemistry—who, by the way, are always most anxious to safeguard the interests of Public Analysts—have taken up this matter very energetically, and have addressed a strong protest to the Lord Mayor of Bristol. Many of those present will be interested to know that the Council of the Institute of Chemistry has recently appointed a committee to consider, among other questions, the appointment of Public Analysts.

I must apologize for having devoted so much time to questions which have a direct interest for Public Analysts only. Some of our most distinguished members do not occupy such positions, and it has been thought by many of us that the scope of the Society, and possibly the name, might be extended so as to make them more representative of the true composition of the Society.

With this object, the question of the incorporation of the Society was raised, and a committee was formed. Several meetings of the Incorporation Committee have been held, and the Rules and the Articles of Association have been drafted and submitted to counsel. Though one would be sorry for sentimental reasons to drop the name which we have honourably assumed for so many years, yet there is much to be said in favour of an alteration. The members of the Society will shortly have an opportunity of expressing their views on the whole matter.

It is intended to hold a meeting of the International Congress of Applied Chemistry in London in 1907, and the Society has been invited to appoint representatives to the joint committee.

In May last it was again brought to the knowledge of the Council that the National Physical Laboratory were in the habit of undertaking chemical analyses for payment in such a way as to compete with the professional chemist. A very strong protest was sent on behalf of the Society, and, as the result of the correspondence and interviews which took place, with the co-operation of the Institute of Chemistry and the Society of Chemical Industry, it is satisfactory to know that Lord Rayleigh, at least, has realized that we are really in earnest in making our protest. He has assured us that in future nothing shall be done to interfere with the rights of private analysts; and though the assurance is couched in somewhat vague terms, we shall be satisfied if the director will see that not only the spirit, but the letter, of the promises are adhered to.

It has also been promised that the Test Pamphlet, which in its present state is, I am bound to say, practically an advertisement, is to be considerably altered. If this is done to our satisfaction, the National Physical Laboratory will have the cordial support of all our members.

I have now briefly referred to the matters which I deemed to be of interest to you. There are one or two other matters which I should have liked to mention, notably the question of standards for whisky, but as this matter is still *sub judice*, it is not proper to discuss it. No doubt the members will have an opportunity before long of expressing their views.

In conclusion, let me again thank you for your courtesy and consideration during my year of office, and for the attention with which you have listened to this somewhat fragmentary address.

* * * * *

ANALYSIS OF A SAMPLE OF AIR WHICH EXTINGUISHES FLAME.

By BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, March* 7, 1906.)

In a large cold storage it was recently found that candle and lamp flames became extinguished, and I was asked to ascertain the cause of this occurrence. Observation on the spot showed that a candle or lamp burning brightly in the outside air began to burn feebly when brought into the storeroom, and in many parts of the room speedily went out. A wax vesta could be struck, and the composition of the head would burn, but the wax would not take fire, and a candle could not be lighted before the match was extinguished. All this happened, although the door of the room was opened at fairly frequent intervals to allow of goods being brought in and out. At a part of the room where flames were most easily extinguished there was the shaft of a disused well, and from this considerable volumes of gas were issuing. This gas extinguished flame with great ease. The natural supposition was that some source of CO_2 was tapped by the well, and that this gas was distributed into the air of the room, producing the effects observed. Samples of the air of the room and of the gas from the well-shaft were collected and analysed, with results which disproved this assumption. It was found that only a trifling amount of CO_2 was present in the air of the room, scarcely more than is present in normal air. It could have no influence on the combustion of a candle. It was also found that all the samples of the air of the room were deficient in oxygen, as will be seen from the following analysis:

		Per Cent. O by Volume.
Sample from	floor near shaft, 12.15 p.m., October 17, 1905 ...	17·6
,,	,, west side centre, 12.25 p.m., October 17, 1905 ...	17·5
,,	,, mid east of store, 2.10 p.m., October 19, 1905 ...	17·6
,,	,, south end, 2.10 p.m., October 19, 1905	17·7

The percentage of oxygen in normal air is 20·9 per cent. by volume. In the four samples cited above there is a deficiency of 3·2 to 3·4 per cent., calculated on the air, corresponding with a deficiency of 15·3 to 16·3 per cent. of the volume of the oxygen itself. It is well known that lights are extinguished in air thus impoverished. That the source of this impoverished air is to be assigned to the gases issuing from the shaft is proved by the analyses given below:

		Per Cent. O by Volume.
Sample from	6 feet down shaft, taken 12.5 p.m., October 17, 1905 ...	8·6
,,	,, 6 feet down shaft, taken 2 p.m., October 19, 1905 ...	8·7
,,	,, 17 feet down shaft, taken 11.30 a.m., October 18, 1905	8·9

A special search was made for combustible gases such as CO, H, CH_4, and other hydrocarbons, both in the air of the room and in that from the shaft, the last named being kindly carried out by my friend Mr. W. J. A. Butterfield. In neither case was indication found of their presence. In fact, the air in the room was simply ordinary air mixed with air depleted of oxygen, which issued from the shaft.

The source of the air coming from the shaft and the cause of its depletion were now sought. Near the cold storage tunnelling operations were in progress, and for these a large supply of compressed air was in use. The air driven into the tunnel finds its way out wherever the ground is sufficiently pervious, and there is little doubt that some found its way to the well, and so up the shaft. In its passage this air is in intimate contact with the soil, and may suffer chemical change. Now, it is noteworthy that the change which has taken place is not of the nature of ordinary oxidation of carbonaceous matter, because in the gas issuing from the shaft there is practically no CO_2. It follows that the substance which has depleted the air of oxygen must be some material the product of whose oxidation is not a gas. Such a substance is pyrites. To test this idea, a sample of mud was taken from another well-shaft near the first, was analysed, and found to contain a notable quantity of pyrites. Further, ordinary air was kept in contact with this mud, and then analysed. It was found to be almost wholly depleted of oxygen. I think it may be fairly concluded that the air coming from the shaft was ordinary air derived from the supply to the tunnel, depleted of oxygen by passage through moist ground containing pyrites.

DISCUSSION.

The PRESIDENT (Mr. Bevan) said that he was rather surprised that the rush of air from the shaft was so violent, and it seemed strange, although the pyrites would be very finely divided, that the air should be able to get into contact with sufficient of it to produce the effect described.

Mr. FAIRLEY said that it would be of interest to know the composition of the soil itself. Mr. Blount was probably correct about the pyrites, though no doubt there would also be a certain amount of organic matter, etc., that might have something to do with the final result.

Mr. SEYLER thought that it was generally admitted that animals could bear with impunity a very much greater deficiency of oxygen than would suffice to bring about the extinction of a candle flame. Some fishes in particular could live when the tension of the oxygen in the water was extraordinarily low, and he fancied that the relative tension of the oxygen in the atmosphere might fall to almost as great an extent as in water without harm to animal life. Ground air was usually deficient in oxygen, but the absence of CO_2 in this case was very striking. The deficiency of oxygen in ground air was probably, as a rule, due to decomposition of carbonaceous matter by fermentation, and consequently ground air was generally fairly rich in CO_2. He had, however, met with a similar case to this, and he thought that it was really not at all uncommon. In different parts of the country bubbling springs or "laughing wells" occurred, which, when disturbed, gave off bubbles of gas. He had analysed the gas from one of these, and found it to be almost pure nitrogen. There was a certain amount of CO_2, but nothing at all equivalent to the oxygen removed. It seemed that in that case the water had fallen on a gathering-ground and percolated through a porous limestone formation, so that the air dragged in with the water was deprived of its oxygen during its passage through the limestone, and nearly pure nitrogen was delivered at the spring. He attributed the abstraction of the oxygen to the action of traces of ferrous compounds in the limestone.

Mr. J. H. B. JENKINS asked how far the well-shaft was from the tunnelling shield, and what depth of soil the air was forced through.

The PRESIDENT asked whether Mr. Blount had taken into consideration the possibility of absorption of oxygen by the sea-water.

Dr. VOELCKER said that the question of the composition of the soil was important, because, if the absorption of oxygen were due to the oxidation of pyrites, a continuous action of that kind must effect a marked change in the constitution of the soil. One would also like to know a little more as to how the shaft lay in relation to the room, its length, the depth and nature of the soil, and so forth. It seemed to him doubtful whether the mere passage of air through a mass of soil collected together as described would really be effective in removing the oxygen to any large extent.

Mr. FAIRLEY said that Mr. Seyler's mention of bubbling springs reminded him of the "blowing wells" which were found in certain parts of this country, and also, he believed, in America. These were blowing either inwards or outwards, according to the state of the barometer, which meant that the shaft of the well was in communication, either above or below the water-level, with large cavities in fissured rocks, particularly in limestone. He had analysed the air coming from some of these "blowing wells," and had found it to be practically the same as ordinary atmospheric air.

Mr. L. MYDDELTON NASH asked if Mr. Blount could say what length of time was required for the removal of the oxygen by the soil in the laboratory experiment.

Mr. BLOUNT, in reply, said that the experiment he had referred to, in which air was confined with the mud in a tube, was not designed to ascertain the minimum time necessary for the removal of the oxygen. The air was allowed to remain in contact with the mud for some weeks, but probably a much shorter time would have been sufficient. In any case, however, such an experiment was a very poor method of reproducing the conditions obtaining when air was blown through the mud and allowed to come intimately into contact with all the fine particles. The President was a little sceptical, perhaps quite justly, as to the possibility of removing the oxygen by so rapid a passage of the air through the mud, but he (Mr. Blount) thought that the oxidation of the pyrites in such a large volume of the mud might well be quite rapid. One must regard the matter in the light of the case of an absorption tube, through which one could pass a large volume of air containing a very small quantity of some removable gas, and still take up that gas perfectly at a very rapid rate of flow. In this case the mass of soil corresponding to the absorbent material of an absorption tube was huge. The section of soil between the end of the shield and the beginning of the shaft was many yards square, and the depletion of oxygen from this cause, to his mind, presented no difficulty.

In reply to a further question put by the President, Mr. BLOUNT said that as far as he knew the temperature was merely the ordinary soil temperature of about 50° F. Continuing, he said that he agreed that a complete analysis of the soil would have been an advantage, though only at the end of the inquiry, when, having discovered the loss of oxygen, he had tried to find out its cause; and the theory as to the pyrites had been accepted as satisfactory for the moment. It was, of course, possible that the conclusion arrived at might be modified in the light of a full proximate analysis of the soil, but he did not think it probable, because the only material likely to be more active than pyrites was ferrous carbonate, and if ferrous carbonate were present and were oxidized, CO_2 would come off, and the results had shown that there was no CO_2. He did not remember the exact distance between the shaft and the shield, but it was not more than a few hundred yards. As to the absorption of oxygen by sea-water, no doubt that might occur, but he thought it would hardly explain the large removal of oxygen that took place in this case. There must be present, somewhere in the mass of soil through which the air passed, a material which would take up oxygen very rapidly, and he thought that nothing but a readily oxidizable substance would be likely to be effective in such a short time. If it were pyrites, it was probably marcasite.

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ON A SIMPLE METHOD FOR THE APPROXIMATE DETERMINATION OF BORIC ACID.

By Cecil H. Cribb, B.Sc. (Lond.), F.I.C., and F. W. F. Arnaud, F.I.C.

(*Read at the Meeting, March* 7, 1906.)

The process about to be described is not intended to replace any of the plans at present in vogue for the determination of boric acid, but has been found very useful as a check on the results obtained by other methods, especially in those cases where the quantity of the sample at the disposal of the analyst is very small.

The Thomson process, as employed for milk, besides being somewhat lengthy, has the further disadvantages of using up a large volume of the sample, and of not being diagnostic of boric acid. The results may be made to vary considerably by slight differences of procedure, and consequently a duplicate determination by the same or some other method is necessary. In many instances the bulk of the sample does not admit of this.

In the case of butter and cream, some plan of rapidly sorting out those samples having more than 0·5 per cent. and 0·25 per cent. respectively of boric acid from those having less is also a desideratum, which even the modification of the Thomson process, proposed by Richmond, does not quite supply.

The method now being brought forward has no claim to superior accuracy, and does not embody any new principle, being based on the well-known colour reaction with turmeric. Several processes have already been published which make use of this, but, as far as our knowledge goes, not one of them has met with general acceptance. That of Cassal and Gerrans (*British Food Journal*, 1902, p. 210) was the first in which any attempt was made to increase the delicacy of the colour reaction, and oxalic acid was added to the turmeric with this object. We found the process as a whole much too lengthy for general use, but the addition of the oxalic acid undoubtedly gave a greatly increased sensitiveness even to turmeric-paper, and the possibilities thereby opened up led us to make further experiments.

Another method based on the change in colour of a turmeric decoction produced by boric acid is that of Hebebrand (*Zeit. f. Untersuch. Nahr. u. Genuss.*, 1902, 55-58), which consists in comparing the colour given by the sample under examination, after, of course, suitable preparation, with that produced under precisely similar circumstances, by known volumes of a standard solution of boric acid. We found, however, that besides wasting a good deal of alcohol and hydrochloric acid, the process was greatly lacking in delicacy.

As we had found that the addition of oxalic acid, as recommended by Cassal and Gerrans, added to the sensitiveness of turmeric-paper as well as to that of the decoction, we prepared papers with a number of other substances, and finally came to the conclusion that tartaric acid gave the best results, and that a paper prepared in the following manner was sufficiently delicate for the end we had in view: Take 2 parts by weight of turmeric and 2 of tartaric acid, and digest with 100 volumes of hot alcohol (about 80 per cent.) until all the tartaric acid has dissolved; then saturate fairly thick blotting-paper with the filtered solution, and hang up to dry, preferably in a dark place.

Paper made in this way turns, with strong solutions of boric acid, a bright rose-pink colour, and the same colour is still just apparent when the dilution is as great as 0·0025 per cent., 2·5 parts per 100,000.

To get the best results it is necessary to pay strict attention to a number of details. The turmeric-paper should preferably be fairly fresh, but we have found it keep its power without deteriorating for as long as a month. It is better preserved in the dark. The degree of acidity of the solution to be tested is of considerable importance. Tartaric turmeric-paper will give excellent results with boric acid alone (but not with borax), although ordinary turmeric hardly changes colour at all under these circumstances. We find, however, that the addition of a small proportion of hydrochloric acid considerably increases the sensitiveness, and in practice we have adopted 2 per cent. as the standard strength. If more is used the paper chars when dried, even at a moderate temperature; and with as little as 2 per cent. the drying operation should be conducted slowly—*i.e.*, at a temperature under 60° C.—and is best done in the dark.

Having prepared a sensitive paper, it only remains to apply it in such a way as to get quantitative results.

A number of possible plans will at once suggest themselves, and we shall content ourselves with giving merely as illustrations the actual method of working which we have adopted (1) as a check on other and more accurate methods in the case of milk, and (2) for controlling the proportion of boric acid in butter and cream.

1. *Milk.*—As the process is mainly intended for use in those cases where the quantity of the sample does not admit of a duplicate determination by the Thomson or other method, we use only a small volume, generally 5 c.c., but even less can be made to do. This quantity is mixed with 1 c.c. of normal alkali, and the whole is evaporated to dryness and incinerated. One c.c. of normal acid is added to the ash, which need not be absolutely white, and the volume is made up to 5, 10, 20, or 30 or

more c.c., according to the proportion indicated by the previous estimation, with 2 per cent. hydrochloric acid. A small strip of paper, 2 inches by ½ inch, is then dipped into the liquid and dried in the dark at a gentle heat. If the paper then exhibits the rose-pink colour, the solution is diluted still further, and each dilution is tested with the turmeric-paper until a stage is reached when it fails to give the characteristic reaction at all. If the degree of sensitiveness is known, it is obvious that a simple calculation will be sufficient to show whether the dilution is what it should be for the indicated percentage of boric acid.

To ascertain the degree of sensitiveness which need only be done at intervals, as the paper changes but slowly, a similar quantity of boric-acid free milk is treated precisely as above, after the addition of a known volume of a 1 per cent. solution of boric acid, and the ash of this is examined by means of the turmeric-paper precisely as already described, and the degree of dilution noted.

To give an idea of the dilution possible, we may instance a recent case of milk which was found to contain, by Thomson's process, 0·119 per cent. of boric acid. Five c.c. of this, when treated in the above manner, bore the addition of 95 c.c. of 2 per cent. hydrochloric acid, and still just showed the colour with the turmeric-paper.

2. In the case of butter and cream, the process is conducted on precisely the same lines. The amount of dilution of the ash of 1 gram of butter containing 0·5 per cent. of boric acid, or cream containing 0·25 per cent., using paper of average delicacy, is about 1,000 times in the case of butter and 500 times in the case of cream. If, therefore, the samples under examination bear a greater dilution than this, they must obviously contain more than 0·5 or 0·25 per cent. of boric acid respectively. Of course, it is in all cases necessary to ascertain the delicacy of the paper by testing it against a standard in the manner already described.

DISCUSSION.

The PRESIDENT said that he did not quite understand the authors' remark that the Thomson process was not diagnostic of boric acid.

Mr. CRIBB said that, although, of course, presumptively the estimation was one of boric acid, it was, strictly speaking, only an estimation of acidity, and slight differences in procedure were sometimes followed by extraordinary differences in the results. It would not do to take the results given by the Thomson process as boric acid without making use of the turmeric reaction, or some similar qualitative test, to see that boric acid was actually there.

The PRESIDENT thought that, provided that boric acid was ascertained really to be present, the figure given by the Thomson process might safely be taken as an accurate representation of the quantity.

Mr. CRIBB said that sometimes there were considerable differences between the results obtained by different operators.

The PRESIDENT said that the figure obtained depended upon the exact point at which the solution ceased to be acid to methyl orange. This was somewhat difficult to determine. He should like to ask whether the increase in sensitiveness which accompanied the use of tartaric acid might not be also obtained by the addition of more hydrochloric acid, the process being in other respects worked in the ordinary way. He had noticed that, if insufficient acid were added, the reaction was not obtained. He could confirm what the authors had said as to the non-keeping properties both of turmeric-paper and of the alcoholic solution.

Mr. CRIBB said that, when more hydrochloric acid was used instead of tartaric acid, it was impossible to dry the paper in a reasonable time without charring it. The colour obtained with turmeric-paper when tartaric acid was present was, moreover, quite different from that given by paper prepared in the ordinary way.

Mr. P. A. E. RICHARDS said that he had found the process valuable for sorting purposes, and very sensitive. As little as 5 grains of boric acid per gallon was easily recognised by it when working on 5 c.c., and he thought that with care even smaller quantities might be detected. It was important, however, to keep the hydrochloric acid at about the strength mentioned—namely, 2 per cent.

Dr. RIDEAL said that Mr. Cribb had been good enough to instruct him in this method a year or so ago, and he had used it with very satisfactory results.

Mr. CRIBB said that the colouring matter produced by the action of boric acid on turmeric seemed to be a more or less definite compound, though he did not think it had ever been fully investigated. They found that when an acid solution of boric acid (as dilute as 0·01 per cent.) was distilled, boric acid could be readily detected in the distillate, but the addition of turmeric decoction to the solution caused the boric acid to be partially held back on distillation.

Mr. F. J. LLOYD thought that it would save much time and would conduce to greater accuracy if the operation of burning with alkali could be dispensed with, the test being simply made by adding hydrochloric acid, filtering, and testing the filtrate.

The PRESIDENT remarked that it was quite easy to detect even as little as 1 or 2 grains of boric acid per gallon in milk without burning, the turmeric being simply applied directly to the milk after acidification.

Mr. CRIBB said that they had tried what Mr. Lloyd suggested. For qualitative purposes the turmeric test could, of course, be employed on the milk direct without further treatment, but to use the reaction quantitatively it was necessary to remove the fat. This meant dilution, precipitation of the proteids, and filtration, and the process at once became too complicated and troublesome.

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THE INFLUENCE OF ORGANIC MATTER ON THE EFFICACY OF CERTAIN DISINFECTANTS.

BY M. WYNTER BLYTH, B.A., B.Sc., F.I.C.

(Read at the Meeting, April 4, 1906.)

THE influence of organic matter on chloride of lime, potassium permanganate, and other oxidizing disinfectants, has been studied by several workers. The phenol class has, however, received no such attention, and the statements that the germicidal power of this or that disinfectant "is maintained, no matter what form of organic matter it is mixed with," appear to have been accepted without question.

In a paper on the "Standardization of Disinfectants" (*Journal Royal Sanitary Institute*, October, 1903, 424), S. Rideal and J. T. Ainslie Walker call attention to the importance, when estimating germicidal value, of keeping the amount of culture in the diluted disinfectants constant, and give an example of the difference produced by increasing the quantity of culture in the dilution, but they do not state what class of disinfectant was used. R. H. Firth and Allan Macfadyen, in a report (*Journal Royal Sanitary Institute*, February, 1906, 17) to the committee appointed by the Royal Sanitary Institute to inquire into the desirability of establishing a standard bacteriological method for determining the efficiency of disinfectants, record some experiments on phenol disinfectants in which germicidal values were determined in fæces and in urine, and H. R. Kenwood and R. T. Hewlett (*Journal Royal Sanitary Institute*, February, 1906, 1) have used an almost identical method. The fæces and urine were used, however, with the object of making the experiments represent in a modified form the conditions which might be met in practical disinfection, and not with the idea of determining the actual influence of organic matter, as neither the weight nor the composition of the fæces and urine are recorded.

In reporting to a committee of the Sanitary Institute early in 1903, I drew particular attention to the influence which even small quantities of organic matter have on the comparative germicidal value of disinfectants of the phenol class, and the experiments recorded here are a continuation of the work begun at that time.

The disinfectants dealt with here are phenol, cresylic acid (a mixture of ortho, meta, and para), Izal, Cyllin, resorcin, and pyrogallic acid.

In attempting to determine the influence of organic matter on these disinfectants, I have thought it best to take a fluid rich in organic matter, and containing such substances as might be met with in practical disinfection, although I do not intend to deal in this paper in any way with practical disinfection.

Such a fluid is milk. Milk has the obvious advantages of containing the three great classes of organic matter—*i.e.*, carbohydrate, fat, and proteid—as well as organo-mineral salts, in the combinations and physical condition in which they have been presented to us by Nature. The organic and mineral matter can not only be determined with great accuracy, but the relative quantities can to a large extent be varied at will.

First, the "carbolic acid coefficients" of the various disinfectants were determined by a modification of the Rideal-Walker method (*Journal Royal Sanitary Institute*, October, 1903, 426), the modification being 6 drops of a twenty-four hours old culture at 37° C. of *B. coli communis*, added to 10 c.c. of the diluted disinfectant, in place of 5 drops of a twenty-four hours old culture of *B. typhosus*, added to 5 c.c. of the diluted disinfectant. The larger quantity of disinfectant was taken for the sake of convenience and accuracy, and the smaller quantity of broth-culture to insure the introduction of as little organic matter as possible. The rest of the details of the Rideal-Walker method were adhered to, except that the temperature during the majority of the experiments was low—*i.e.*, 12° C. This temperature gives low results with at least one disinfectant, but does not affect the comparative results with organic matter. Having determined the carbolic coefficients in water, the coefficients were determined in 5 c.c. of milk diluted to 10 c.c. with various dilutions of the disinfectants. The milk used was both milk with all its cream and separated milk, and the composition of each batch was determined.

The milk used in the results given below had an average composition of—

	Fat.	Milk-sugar.	Proteids.	Ash.
Full milk	3·6	4·7	3·4	0·74
Separated milk ...	0·3	4·8	3·6	0·78

Disinfectant.	Carbolic Acid Coefficients at 12° C.				
	In Water (Phenol = 1).	In 50 per Cent. Full Milk (Phenol in Water = 1).	In 50 per Cent. Separated Milk (Phenol in Water = 1).	In 50 per Cent. Full Milk (Phenol in Full Milk = 1).	In 50 per Cent. Separated Milk (Phenol in Separated Milk = 1).
Phenol	1·0	0·8	0·9	1·0	1·0
Cresylic acid ...	2·0	1·5	1·8	1·9	2·0
Pyrogallic acid ...	0·22	—	0·18	—	0·2
Resorcin	0·30	—	0·22	—	0·27
Izal*	7·0	0·75	1·2	0·9	1·3
Cyllin*	5·5	0·75	1·5	0·9	1·6

It will be seen from these results that the constituents of milk have a very marked influence on the germicidal value of the phenol disinfectants, and further experiments showed that the lowering in comparative and actual germicidal value is proportional to the amount of milk constituents present. Thus, in some experiments with Izal a solution of 1 of Izal in 100 water produced sterility in ten minutes when 18 per cent. of milk was present, but not when 36 per cent. of milk was present; 1 : 66 Izal produced sterility in ten minutes with 36 per cent. of milk, but not with 72 per cent.; 1 : 50 Izal produced sterility in thirty minutes with 72 per cent. of milk, but not with 85 per cent. The influence of milk constituents on phenol itself is not nearly so marked as on the cresols and those disinfectants containing the higher phenols, so that as we introduce more milk into the dilutions we get a progressive fall in the comparative carbolic acid coefficient.

The Influence of Fat.—If we compare the coefficients in full milk and separated milk, it will be seen that the introduction of fat causes, according to the disinfectant, a more or less pronounced fall in the coefficient. When separated milk is used, we have slightly more of each organic constituent present except fat and a little more mineral matter than we have when full milk is used. So that if fat has no influence we should expect the coefficient in separated milk to be lower than in full milk; but the reverse is found to be the case. It was further found that when a milk rich in fat was taken, a lower coefficient was obtained than with the milk containing less fat. Thus with Izal a coefficient (phenol in water = 1) of 0·8 was obtained with a milk containing 3·5 per cent. of fat and a coefficient of 0·5 (phenol in water = 1) with 4·2 per cent. of fat.

The influence of fat is probably purely physical, some of the emulsified oils in cresylic acid, Izal, and Cyllin being dissolved by the fat, and removed from contact with the organism. This view is supported by the fact that a 1 per cent. solution of Cyllin, after shaking up with one-third its volume of olive oil, gave a coefficient of *under* 3 at 20° C. (phenol in water = 1), as compared with an original coefficient of 7·7 at 20° C.

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* The proprietary names of two disinfectants on the market.

The Influence of Milk-Sugar.—The coefficients of the disinfectants were taken in 2·5 per cent. of milk-sugar. Although in some cases there was a slight lowering of the coefficient, yet the effect of this quantity of milk-sugar is so small that it may be neglected.

The Influence of the Mineral Constituents.—The ash from 5 c.c. of burnt-up milk had apparently, when shaken up with 10 c.c. of sterile water, no appreciable influence on the coefficients. This experiment, however, proves little, since the constituents of the ash are not in the same combinations as in the natural milk.

The Influence of Proteids.—The only constituents of the milk left which are likely to influence the coefficients are the casein and albumin. If we admit that the milk-sugar and ash have little, if any, influence, and that the fat only accounts for a portion of the loss, we must conclude that the proteids are the main factor in the lowering of the coefficients. This view is supported by the fact that in a 1 per cent. solution of serum-albumin the coefficient of Cyllin was found to be under 3 at 12° C. And in some experiments on gelatin solutions the presence of 2·5 per cent. of gelatin reduced the coefficient of Izal, taken at 40° C., from 7 (in water) to 4·5 (in gelatin. Phenol in water at 40° C. = 1). In the same way with gelatin the coefficient of Cyllin was lowered from 8·4 to 5·3.

Temperature.—In connection with the determination of the coefficients in gelatine at 40° C., the influence of temperature on the three disinfectants, phenol, Izal, and Cyllin, was determined, when an interesting difference was observed. All of them are naturally more active at the higher temperatures, but phenol and Izal increase in activity in much the same ratio, with the result that the coefficient of Izal remains at about 7 over a wide range of temperature. On the other hand, the activity of Cyllin increases, especially at first, more rapidly than that of phenol; consequently there is a rapid rise in the coefficient.

Carbolic acid coefficients of Cyllin at various temperatures:

Temperature ...	8° C.	12° C.	20° C.	40° C.
Coefficient ...	5	5·5	7·7	8·4

These differences are probably due to the variations in the size of the oil globules at different temperatures, Izal, being supplied in the form of a fine emulsion, not being influenced to any great extent by variations in temperature.

Poisonous Properties and Germicidal Value.—Although it is easy to understand how a substance may be fatal to a highly complex animal organism and yet harmless to vegetable organisms, it is not so easy to understand why substances such as Izal and Cyllin are so deadly to "naked" micro-organisms in water and comparatively harmless to animal life, while phenol is much less fatal to naked micro-organisms and yet very toxic to animals.

There seems to be a certain amount of confusion in literature as to the relative poisonous properties of phenol, the cresols, and the di- and tri-hydroxyl phenols. Thus I find it variously stated that resorcin is more poisonous than phenol, and that it can be taken in relatively large doses. Pyrogallic acid is stated to be more poisonous than phenol, but I find that it is considerably less poisonous to small animals, which can be immersed in the liquid. According to Karl Tollen's (*Archiv f. experiment. Path. u. Pharm.*, Bd. ii., S. 239) experiments on cats, mice, and frogs, meta-cresol is less poisonous and para-cresol more poisonous than phenol, while ortho-cresol is as poisonous as phenol to cats and mice, but less so to frogs. For the purpose of seeing if there is any connection in the phenol class between poisonous properties and germicidal value, instead of taking the action on man or large animals, it is, I think, better to take some small organism, such as a worm (*Lumbricus herculeus*), which can be placed under the same conditions as the micro-organisms.

In some experiments with worms of equal weight, I found that the time taken to kill them by means of 5 per cent. solutions was as follows:

Phenol	½ minute.
Cresylic acid (ortho-, meta-, and para-) ...	¾ minute.
Resorcin	3 minutes.
Pyrogallic acid	5 minutes.
Izal and Cyllin	Over 10 minutes.

Taking phenol, resorcin, and pyrogallic acid, the poisonous nature seems to be decreased as we introduce hydroxyl groups, and is directly proportional to their germicidal value. Further, these compounds suffer very little loss in germicidal value in the presence of albumin. Taking phenol, cresylic acid, Izal, and Cyllin, their germicidal value appears to be in inverse proportion to their poisonous nature; but phenol and cresylic acid, which lose but little germicidal value when mixed with milk, are highly toxic; while Izal and Cyllin, which lose about 80 per cent. of their germicidal value in milk, are but little toxic. Both the toxic and germicidal action of these compounds may be explained if we consider that the higher phenols act on the delicate "naked" organisms and unite with the "labile" albumin of the protoplasm, the act of union killing the organism, and converting the albumin into a more or less non-toxic compound. If other albumin is present, a certain quantity of the higher phenols unite with this, leaving less poison to unite with the "labile" albumin. The albumin-phenol compound formed would appear to be more poisonous as we descend from Izal to phenol, and possibly equally poisonous in the mono-, di-, and tri-hydroxy phenols; and it is due to this fact that these last-mentioned bodies maintain their germicidal value in the presence of organic matter. The supposition that a poisonous albumin-phenol compound is formed receives support from the fact that, although resorcin gives a copious precipitate with albumin, it suffers but little loss of germicidal value.

Summary.—Disinfectants containing the higher phenols suffer great loss of efficacy when mixed with (1) fat, (2) albumin, and (3) fæces and urine (see Kenwood and Hewlett, *Public Health*, February, 1906, and April, 1906).

The germicidal value of a disinfectant acting on a "naked" organism gives little, if any, indication of its value in the presence of organic matter.

It appears impossible in the phenol class to combine a low toxic value with a high germicidal value in the presence of much organic matter.

DISCUSSION.

The PRESIDENT (Mr. Bevan) having invited discussion,

Professor KENWOOD said that Mr. Wynter Blyth's paper afforded further evidence of the insufficiency of any method which aimed at obtaining the germicidal value of a particular preparation when that method dealt with naked organisms, for it demonstrated how largely the introduction of organic matter affected the case at issue. It was that very circumstance that made necessary the revision of most of the conclusions based on the work on disinfectants during the last fifteen or twenty years; for a very large proportion of that work had either been performed upon naked organisms, or had been carried out according to methods which at the present day were open to criticism. The author had referred to Messrs. Rideal and Walker's method of standardizing disinfectants, and had said that in his opinion the flaw in that method was the fact that it did not take into account the effect which the associated organic matter had upon the disinfectant. With that opinion he (the speaker) and many others were in agreement. By that method it had been shown that potassium permanganate was about the most efficient disinfecting agent possible, whereas it was well known that when a large amount of organic matter was present the use of permanganate as a germicidal agent was unsatisfactory, because it acted preferentially on the dead (and more especially the putrescent) organic matter, and was therefore often used up by associated dead organic matter before the living germs were destroyed. Although, however, the Rideal-Walker method suffered from that defect, he, in common, he believed, with Mr. Wynter Blyth, should like to express his appreciation of a valuable suggestion, for he thought that on the lines indicated by Messrs. Rideal and Walker a method of standardization might be arrived at which would take into account the effects produced by the association of the germ with organic matter. The method, however, as originally proposed, and as applied at any rate up to the present, was certainly defective and unacceptable. The results of his own experiments, in conjunction with Professor Hewlett, upon typhoid fæces from one of the large fever hospitals in London were quite in accord with those obtained by Mr. Wynter Blyth. They had found that the coefficients obtained with the naked organisms were totally different from those obtained when they sought to attack the germ in its usual environment of a relatively enormous quantity of organic matter. Their difficulty had been to find a standard medium in which the organisms could be planted, and which would present the average difficulty of attack which is encountered when any of the natural media of fæces, urine, sputum, pus, etc., was present. The objections to working with fæces and pus (with the latter of which he was now making some experiments) were, of course, considerable, and he believed that really it was not necessary to use such media as these, but that it ought to be possible to devise a standard medium presenting similar obstacles to attack by disinfectants. The author had pointed out that milk as a standard medium afforded the advantage that its exact composition could be readily ascertained. Certainly milk presented some advantages in this respect, but probably Mr. Wynter Blyth would agree that it would not be entirely satisfactory as a standard organic medium. Its levelling effect was too great, because it was greater than that of fæces, and greater, he believed it would be found, than that of pus. The experimental results given in the paper were very interesting, and he should like, in conclusion, to say that the author had asked him, as his results were rather startling, to check one or two of them. After considerable experience of the action of these disinfectants, he was certainly surprised to hear that the effect of adding the organisms to milk with 50 per cent. of water was to reduce their germicidal coefficients so largely; but on repeating the author's experiment with Izal in comparison with phenol, working with the typhoid germ, he found the carbolic acid coefficient of Izal (which was about 9 by the Rideal-Walker method) to be exactly 1. That was practically in agreement with the author's figure. He had no doubt that the results obtained with regard to the other disinfectants were quite correct, though he had not had time to repeat the author's experiments with these.

Dr. Rideal said that when the relative value of disinfectants was determined in a medium containing organic matter the differences shown between them were much smaller than those shown when water only was used. In other words, in the presence of organic matter the disinfectants were not differentiated from one another so markedly as in the absence of organic matter. The test which Mr. Walker and he had suggested was put forward as a method for *separating* the disinfectants from one another—not for bringing them close to one another; and it was not put forward as a method for determining their relative value for any specific purpose. It might, of course, be said that disinfectants as a rule were used in presence of organic matter. They were seldom used, however, in milk; so that from that point of view milk was just as objectionable as water, and he ventured to think that this applied even to urine and fæces, for it was seldom, after all, that these had to be disinfected by chemical means. In comparing disinfectants from a particular point of view, however, the first thing to be done was to find out how far apart or how close together the different disinfectants were in some neutral, uniform medium, and they had chosen water as a definite thing that could be reproduced in any laboratory. Every chemist, of course, knew without any bacteriological test that potassium permanganate, chlorine, bromine, and so on, were useless in presence of large quantities of organic matter; but it must also be recollected that disinfectants were required in cases where there was not a very large amount of organic matter, such, for instance, as the very important problem of the sterilization or disinfection of water-supplies; and it would be absurd to say that because bleaching powder showed a low germicidal value when tested in fæces or in milk, it therefore would be useless for disinfecting the water-supply in a case like that of the recent typhoid epidemic at Lincoln. It seemed to him, therefore, that even greater objections might be urged against the media suggested by Professor Kenwood and Mr. Wynter Blyth than against water, until one knew what the disinfectant was going to be used for. The question of the toxic value of disinfectants was a very interesting one, but, having regard to the enormous differences which appeared to exist between the effect on germs and on worms, and to the still greater differences which presumably would be found when the effects on human beings were considered, he should not himself like to accept the results of Mr. Wynter Blyth's experiments on worms as indicating either the relative value or the poisonous nature of the disinfectants he had experimented with. With regard to the effect of temperature, Mr. Walker and he had found that as a rule the coefficient rose when the temperature was increased.

Mr. Wynter Blyth said that he had not intended in any way to attack the Rideal-Walker method, but simply to put down the results of his experiments, which showed that when these disinfectants were mixed, not only with milk, but with albumin, gelatin, and fat, the coefficients were considerably lowered. He did not suggest for a moment the adoption of a standard medium in the form of milk. He had taken milk because he thought that, as it was easily analysed and could be varied in composition at will, it would give some idea of the effect of each constituent. He thought that the constituent which had the greatest influence was the albumin, and surely albumin was a body which had to be dealt with almost invariably in practical disinfection. He must say that he failed to understand what was meant when Dr. Rideal said that the Rideal-Walker method was for the purpose of separating disinfectants. Did he mean that it was intended to mislead people as to their true value? With regard to the toxic values, it perhaps would have been better to use the various solutions in exactly the same way as in determining the germicidal values, so as to ascertain how much of each was required to do the same amount of work. That would have given their toxic values as applied to worms. These figures, however, did give some idea of the toxic values as applied to a living animal immersed in the liquid under the same conditions as a micro-organism. Some of the statements on this point in the books were very conflicting. For instance, he found that in Dr. Rideal's book resorcin was stated to be not at all poisonous even in large doses, while in his father's book on "Poisons" it was said to be very much more poisonous than phenol. He had therefore had to make experiments on his own account, as he wanted to trace, if possible, some connection between germicidal value and toxic value. He thought that some such connection must exist, but the point required much further investigation.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Hydrogen Peroxide in Milk. P. Adam. (*Journ. Pharm. Chim.*, 1906, xxiii., 273-277.)—It has frequently been shown that hydrogen peroxide added in small quantity to milk disappears after about six to eight hours, but hitherto no tests have been devised to determine whether a sample of milk ever contained it. The author's methods have been based upon his observations that the different enzymes in the milk differ in their power of resistance to the action of hydrogen peroxide, and that the reducing enzymes are attacked first. Schardinger's reagent (5 c.c. of a concentrated alcoholic solution of methylene blue, 5 c.c. of formaldehyde, and 190 c.c. of water) is invariably decolourized by a milk that has never contained hydrogen peroxide, whatever its age may be; but a milk that has once been so treated loses its power of decolourizing the reagent, and only regains it after about five days, when bacterial decomposition has commenced. A raw, fresh milk that has not been treated with hydrogen peroxide becomes garnet-red on the addition of guaiacol and hydrogen peroxide, and blue with paraphenylene-diamine and hydrogen peroxide. The same milk altered by incipient decomposition does not give these reactions, but still decolourizes Schardinger's reagent. A raw milk containing hydrogen peroxide gives a coloration with guaiacol or paraphenylene-diamine by themselves. A milk from which the hydrogen peroxide has disappeared behaves like ordinary milk in the tests with guaiacol and paraphenylene-diamine, but, as was shown above, does not decolourize Schardinger's methylene blue reagent. A boiled milk does not give any of these reactions. C. A. M.

On the Baudouin Reaction in Human Milk-fat. Engel. (*Zeits. angew. Chem.*, 1906, xix., 283.)—The author examined the milk obtained from three wet-nurses after giving them fairly largely doses (100 grams at a time) of sesame oil. He found that the fat always gave the Baudouin reaction after an interval of only one and a half hours between the feeding and taking the sample, a longer time being needed before the iodine value increased. He also found that when the sesame oil was taken only once, or only for a few days, the fat gave the reaction for the whole period of time during which the iodine number reached its maximum, and that then an interval occurred during which no reaction could be obtained, followed by a third period, on the same day as the second, during which the reaction again occurred. If, however, sesame oil is administered over longer periods, these three intervals lose their distinctness, and the reaction is obtained even after the iodine value has begun to decrease. A. G. L.

A Method for the Detection of Cocoanut Oil in Butter. H. P. Wijsman and J. J. Reijst. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 267-271.)—The method is based on the comparative insolubility of silver caprylate, and consists in precipitating the neutralized distillate obtained in the ordinary Reichert-Meissl process with silver nitrate and determining the amount of silver used. A second distillation on a separate 5 grams of the sample is then carried out, but in this case 300 c.c. of distillate are collected, water being added to the flask during the distillation. If the sample consist of pure butter, the caprylic acid present will all distil over in the first distillation, and the quantity of silver required to precipitate the second distillate will not be more than that required for the first—in fact, rather less, owing to the slight solubility of the silver caprylate in the larger volume of distillate. In the case of cocoanut oil the continued distillation causes a larger amount of caprylic acid to distil over, and the distillate consequently consumes more silver than is required to precipitate the first distillate.

The actual details of the method are as follows: Five grams of the sample are saponified with sodium hydroxide and glycerol, and the Reichert-Meissl value determined as usual. Forty c.c. of $\frac{N}{10}$ silver nitrate solution are then added to the neutralized filtrate, the precipitate is collected on a filter and washed, until about 200 c.c. of filtrate have been collected. Fifty c.c. of $\frac{N}{10}$ sodium chloride solution are added, and the excess of chloride titrated back with the silver solution, using potassium chromate as indicator. The number of c.c. of silver solution used when increased by one-tenth is termed the "first silver value."

Another 5 grams of the sample are similarly saponified and distilled in the usual way. When 100 c.c. of distillate have been collected, 100 c.c. of water are added by means of a tapped funnel to the flask and the distillation continued. This addition of water is once more repeated. The 300 c.c. of distillate are mixed, filtered, and 250 c.c. of the filtrate titrated as in the Reichert-Meissl process. To the neutralized solution 40 c.c. of $\frac{N}{10}$ silver nitrate solution are added, the precipitate is filtered off, washed to yield about 350 c.c. of filtrate, 50 c.c. of $\frac{N}{10}$ sodium chloride solution are added, and the excess titrated as described above. The number of c.c. of silver solution used increased by one-fifth constitutes the "second silver value." If the latter is higher than the first silver value, cocoanut oil is present in the sample under examination. In the case of pure butter, with a Reichert-Meissl value of 28·4, the first and second silver values are shown to be 5·9. The addition of 5 per cent. of cocoanut oil to this butter raised the values to 6·4 and 7·3 respectively. W. P. S.

Detection of Horse and Fœtal Flesh by means of the Glycogen Determination. M. Martin. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 249-266.)—The glycogen occurring in horse and fœtal flesh remains unaltered for a long time, whilst in beef, veal, or pork only traces, if any, can be detected a few days after the animal has been slaughtered. It is therefore possible to detect the presence of horse or fœtal flesh in sausages, etc., by a determination of the glycogen, for which purpose the original method of Pflüger (*Pflüger's Archiv*, 1902, xxii., p. 163) gives the best results. In this method the flesh to be examined is digested with potassium hydroxide and the sugar determined in the solution (*cf.* ANALYST, 1903, p. 320). The sample should be kept for a few days before the analysis is carried out, and if any glycogen be then found horse-flesh is very probably present; but other sources of glycogen (starch, etc.) must be taken into account. The glycogen in smoked or potted horse-flesh gradually disappears, and the determination is therefore futile in such articles. The unusual stability of the glycogen in horse-flesh may be due to the feeble fermentative action of horse-blood as compared with ox-blood.

W. P. S.

A Study of the Phosphorus Content of Flesh. A. D. Emmett and H. S. Grindley. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 25-63.)—The investigation recorded had for its object the determination of the quantities, the distribution, and the nature of the inorganic and organic phosphorus compounds occurring in the different species of animals the flesh of which is used as food for man. The results obtained in the cases of beef and veal are given. The Hart-Andrews method (ANALYST, 1904, p. 97) of separating and determining the inorganic and organic phosphorus was found to give satisfactory results in aqueous extracts of flesh after the coagulable proteids had been removed. From the figures given it is seen that there is a difference in the phosphorus content of the flesh of beef and veal. Of the total phosphorus in beef, 75 per cent., and in veal, 64 per cent., is soluble in cold water. In beef one-fourth of the total phosphorus is present as soluble organic phosphorus and in veal one-sixth, whilst the soluble organic phosphorus in beef constitutes one-third of the total soluble phosphorus and one-fourth in veal. The ratio of the soluble organic to the soluble inorganic phosphorus in beef is 3 : 5, and in veal 3 : 9. The ash of beef contains 23·4 per cent. of phosphorus and the ash of veal 20·2 per cent. The soluble forms of phosphorus in beef constitute 17·8 per cent., and in veal 12·8 per cent., of the ash. The percentage of fat in the different cuts of veal has little influence on the total phosphorus content, but the cuts which are nearest the bony structure apparently contain more insoluble phosphorus than the other cuts. Different methods of cooking flesh give products which vary decidedly as to the quantities and nature of the phosphorus compounds they contain. The water-soluble organic phosphorus of the aqueous extracts of flesh is not in combination with the coagulated proteid, with the albumoses, or with the peptones; in other words, the proteids of aqueous extracts of flesh do not contain phosphorus, but the organic phosphorus of such solutions is due to the presence of non-proteid bodies. The soluble organic phosphorus compounds in flesh are quite stable, even in the presence of a considerable excess of nitric acid (see also ANALYST, 1905, p. 89). W. P. S.

Artificial Coloration of Preserved Truffles. Frehse. (*Ann. de Chim. anal.*, 1906, vol. xi., p. 98.)—A sample of preserved truffles examined by the author was found to have been artificially coloured by means of tannin and a salt of iron. The interior of the fungi was soft, and yielded to the touch, instead of being elastic, as in the case of good truffles. The white, sinuous veins of the normal product were hardly perceptible, though they became more visible on treatment with hydrochloric acid. The following comparative results were obtained in the analysis of the interior portion of an ordinary black truffle and of this sample :

	Water.	Ash.	Insoluble Ash.	Ash Insoluble in HCl.	Alkalinity of Ash (in terms of H_2SO_4).	Chlorine as Sodium Chloride.	Iron Oxide.
Black truffle ...	77·56	1·58	0·62	0·01	0·41	Trace	None
Preserved truffle ...	83·04	1·19	0·50	0·057	0·16	0·29	0·05

On treating the inside of the dyed sample with hydrochloric acid, extracting the liquid with ether, and evaporating the ethereal extract, a black residue, consisting of an iron tannate, was left. C. A. M.

Determination of Mercury and Iodine in Antiseptic Soaps. A. Seidell. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 73.)—A weighed amount of the soap is dissolved in about 100 c.c. of 95 per cent. alcohol, the solution acidified with 3 to 5 c.c. of concentrated hydrochloric acid, and warmed, successive small quantities of water being added until a perfectly clear solution is obtained on shaking, any suspended matter being filtered off. Sulphuretted hydrogen is then passed slowly through the liquid for about an hour, the precipitated mercuric sulphide filtered on a Gooch crucible, dried, and weighed. Only the least suction should be used until the sulphide has been washed several times with 95 per cent. alcohol, as the precipitate tends to pass through the filter, due to the fatty matter in solution. The filtrate and washings are concentrated on the water-bath to about one-half the original volume, water added to replace the alcohol, the solution cooled, and the fatty acids filtered off. The clear liquid is then transferred to a separating funnel, the iodine liberated by addition of a few drops of nitrous acid, and dissolved out by extracting two or three times with 25 to 50 c.c. of chloroform. The extract, after washing with distilled water, is finally titrated with standard thiosulphate solution, the washings being treated with fresh chloroform to remove any dissolved iodine. The nitrous acid solution is prepared by treating a mixture of 10 grams each of starch and arsenious acid with 150 c.c. of nitric acid (specific gravity 1·3), the gases evolved from the solution on warming being led into a bottle containing about 100 c.c. of concentrated sulphuric acid. W. H. S.

The Determination of Iodine in Iodvasogen and Similar Preparations. C. Arnold and G. Werner. (*Pharm. Zeit.*, 1906, li., 84; through *Chem. Zeit.*, 1906, xxx [*Rep.*], 54.) Pringsheim's method (ANALYST, xxix. 97). About 0·5 gram of the substance is mixed with 9 to 10 grams of sodium peroxide in an iron crucible, which is placed in a basin of water, whilst the contents are ignited by introducing a hot wire through an opening in the crucible lid. The reaction is complete in a few minutes, and the crucible is then washed out into the basin, the liquid filtered, and the filtrate and washings rendered slightly acid by the addition of dilute sulphuric acid containing about 2 grams of sodium sulphite in solution. The sulphurous acid reduces the sodium iodate to iodide, which is determined gravimetrically by treating the liquid with nitric acid and adding silver nitrate. C. A. M.

Valuation of Pepsin by Means of the Biuret Reaction. W. B. Cowie and W. Dickson. (*Pharm. Journ.*, 1906, lxxvi., 221-223.)—The biuret test, consisting in the addition of copper sulphate and potassium hydroxide to a peptone solution, gives a coloration which increases in intensity as the quantity of peptone present is augmented, and on this reaction the authors base a method for the determination of the amount of peptone produced by a sample of pepsin when the latter is allowed to act on albumin. A quantity of coagulated egg albumin equivalent to 1 gram of dry albumin is triturated with 10 c.c. of water, and washed into a 100 c.c. flask with a further 10 c.c. of water. The flask and its contents are placed on a boiling water-bath for fifteen minutes, then cooled to a temperature of 40° C., and 0·25 gram of the pepsin under examination, together with 25 c.c. of $\frac{N}{10}$ hydrochloric acid, are added. The mixture is kept at a temperature of 40° C. for four hours, the contents of the flask being shaken every half-hour. At the end of that time the mixture is heated to boiling, cooled and diluted with water to 100 c.c. Ten c.c. of the liquid are now treated in a test-tube with 13 grams of zinc sulphate and 0·2 c.c. of dilute sulphuric acid (1 : 4), the whole heated to boiling, then quickly cooled and filtered. Five c.c. of the filtrate are placed in a Nessler cylinder, mixed with 15 c.c. of water and 1 c.c. of 0·5 per cent. copper sulphate solution, and the volume made up to 80 c.c. with 30 per cent. sodium hydroxide solution. Seventy-five c.c. of water are placed in a second cylinder, and a potassium permanganate solution (containing 0·04 gram per litre) run in until the depth of colour is the same in both cylinders. In the following table are given the results obtained with two samples of pepsin, four digestions with each sample being put in process.

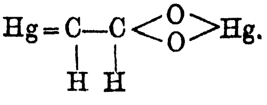
Time of Digestion.	Sample 1. C.c. of $KMnO_4$ required.	Sample 2. C.c. of $KMnO_4$ required
One hour	4·0	2·1
Two hours	5·0	2·7
Three hours	5·5	3·5
Four hours	6·0	3·7

A blank experiment showed that the pepsin No. 1 itself indicated peptone after four hours digestion equivalent to 0·9 c.c. of the permanganate solution, whilst sample No. 2 indicated peptone equal to 0·5 c.c. These quantities must be deducted from the figures in the table to give comparative results. It will be seen that the activity of the two pepsins varied considerably from hour to hour. W. P. S.

ORGANIC ANALYSIS.

The Differentiation of Acetaldehyde and Formaldehyde. A. Leys.

(*Ann. de Chim. anal.*, 1906, vol. xi., pp. 84-88.)—Mercuric oxide dissolved in a dilute solution of sodium sulphite forms a double sulphite, which is not precipitated by alkalies. In the presence of a trace of acetaldehyde, however, the addition of a cold alkaline solution produces, after a few seconds, an abundant dense white precipitate. Similar compounds are formed under these conditions by all aldehydes containing the group CH_2-COH; whereas formaldehyde, furfural, aldoses, and certain aromatic aldehydes give only a fine deposit of metallic mercury. The precipitate given by acetaldehyde is produced more rapidly on heating. It is necessary to have the aldehyde solution sufficiently dilute, and to avoid the addition of too much alkali, in order to prevent complete reduction, with the formation of a deposit of metallic mercury. The compound formed is a white powder, insoluble in water, alcohol, and boiling dilute sulphuric acid, but readily soluble in a dilute solution of potassium cyanide, and with difficulty in hot dilute nitric acid. It also dissolves easily in dilute hydrochloric acid, the solution emitting a pungent odour when heated. Its elementary composition and properties correspond with the formula:



$$\mathrm{Hg{=}\underset{H}{\underset{|}{C}}{-}\underset{H}{\underset{|}{C}}<{}^{O}_{O}>Hg.}$$

Of the better-known salts of mercury, only mercuric chloride can replace the oxide in the preparation of the reagent. The precipitate formed with acetaldehyde then contains a certain proportion of chlorine.

The reagent is best prepared by gently heating 1 gram of powdered mercuric oxide with 100 c.c. of a recently prepared 5 per cent. solution of sodium sulphite, allowing the solution to cool, and filtering it from the small deposit of reduced mercury, with precautions to prevent contact with ammonia vapour (even in the atmosphere). In making a test, 10 c.c. of the reagent are treated with a few drops of the aldehyde solution, and then with an equal volume of the alkaline solution. A certain proportion of alcohol may be added without interfering with the reaction, but when this quantity is exceeded the reagent is precipitated as a crystalline compound. Acetone gives no reaction in the cold, but on heating the liquid a white precipitate resembling that formed with acetaldehyde is produced. Acetyl-acetone gives an abundant deposit, even in the cold, after the mixture has been shaken for some time. Acids containing an ethylene group—*e.g.*, oleic, malic, and crotonic acids—give no preciptate, whilst others, such as formic acid and tartronic acid, give only a deposit of mercury. C. A. M.

A New Method of Determining Acetone in Urine. F. Bluth. (*Deutsch Med. Woch.*, 1906, xxxii., 143; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 69.)—The method is based upon the fact that the time required for the red coloration given by urine containing acetone with sodium hydroxide and sodium nitroprusside solution to change to orange or yellowish green is, within certain limits, proportional to the amount of acetone. The bulk of the creatinine is first removed by means of zinc chloride, and the urine decolourized with lead acetate. A correction is subsequently made for the residual creatinine by repeating the determination after removal of the acetone from the urine by evaporation, and deducting the time then required for the change of colour. Aceto-acetic acid has a very great influence on the speed of the reaction, and when it is present the urine must be boiled for five minutes in order to decompose the acid into acetone and carbon dioxide, the vapours evolved being collected in cold water. C. A. M.

Method for the Determination of Pentosans. A. Jolles. (*Monatsh. Chem.*, 1906, xxvii., 81-90.)—In the method described the vegetable substance is distilled with hydrochloric acid, and the furfural formed titrated with sodium hydrogen sulphite solution. The details of the process are as follows: From 0·2 to 1 gram of pentose, or the corresponding quantity of substance, is placed in a round-bottomed flask, A, of about 1½ litres capacity, and 200 c.c. of HCl (specific gravity 1·06) added. In a second similarly sized flask 900 c.c. of H_2O are placed and heated to boiling. The steam from this is led to the bottom of flask A by means of a bent tube passing through one aperture of a doubly perforated indiarubber cork, while through the second aperture another tube leads to a Liebig's condenser. With this apparatus the whole of the furfural yielded completely passes over with the steam. The distillation is to be so carried on that during the time in which from 700 to 800 c.c. of water have evaporated in flask B the volume of liquid in flask A does does not diminish more than about 100 c.c., which condition may be easily secured by regulating the gas supply to the burner. After cooling, 50 c.c. HCl of specific gravity of 1·06 are added to flask A, about 800 c.c. H_2O to flask B, and the process repeated. The repetition is continued until 1 c.c. of the distillate with 4 c.c. of Bial's reagent (a solution of 1 gram of orcinol in 500 c.c. of concentrated HCl, to which is added 20 drops of a 10 per cent. ferric chloride solution) shows no reaction on boiling. As a rule the decomposition is complete when from 2,000 to 3,000 c.c. have passed over into the receiver. The distillate must be well agitated to insure perfect admixture, and measured at 15° C. A measured quantity (100 c.c.) of the distillate is neutralized (under cooling) with 20 per cent. NaHO solution, using methyl-orange as indicator, about 2 drops of KHO solution added, then titrated with $\frac{N}{2}$ HCl until a rose colour appears. After standing a few minutes the colour changes to yellow, when 2 drops of HCl are added, and the whole allowed to stand for a few minutes to see if the colour again changes; this is repeated again and again until the colour remains permanent. Two hundred c.c. of H_2O are added, a measured quantity (at least 20 c.c.) of an approximately $\frac{N}{5}$ or $\frac{N}{10}$ solution of $KHSO_3$ mixed in, and after standing for two hours, the excess of bisulphite ascertained by iodine solution. In each analysis the titre of the bisulphite solution should be standardized against the same I solution, which has been used for the back titration. In this and in the titration itself stoppered bottles must be used, as also in the standardization; the bisulphite solution is diluted and allowed to stand two hours, so that its instability may be taken into account. The burette used should be graduated in twentieths of a c.c., and is most readily read by means of a float and lens.

The titration of the furfural cannot be performed in an acid distillate, nor in one containing an excessive quantity of salts, hence the steam distillation described above. An acidity amounting to 3 drops of $\frac{N}{2}$ hydrochloric acid is, however, without influence on the results.

In calculating the result, one molecule of pentose equals one of furfurol, and these are equivalent to one molecule bisulphite, or two atoms of I; therefore the quantity of bisulphite used is to be converted into c.c. of normal solution; 1 c.c. N-bisulphite solution equals 0·07505 gram pentose—*i.e.*, half a molecule weight.

Experiments with pure furfural and with arabinose show that the method is trustworthy, maximum and minimum differences being +0·8, −0·3 per cent.

W. P. S.

An Adulterant of Lycopodium. C. Gallois. (*Journ. Pharm. Chim.*, 1906, xxiii., 242-244.)—For some time past a powder closely resembling lycopodium in appearance and properties has been sold at a much lower price under the name of *substitute.* It differs from lycopodium in being partially soluble in alcohol, chloroform and ether, the soluble matter being a resinous substance resembling colophony. Under the microscope it is seen to consist of irregular transparent granules. On ignition, it leaves a reddish ash, consisting, in the main, of ferric oxide. In one case this amounted to 2·078 per cent. of the original powder. In the author's opinion, the substance is prepared by the action of anhydrous ammonia upon Austrian (?) galipot resin, dried at a temperature as near as possible to its melting-point. The product is sold at 4 to 4.50 francs per kilo., whilst lycopodium powder has recently fetched as much as 10 to 12 francs per kilo.

C. A. M.

On the Rancidity of Cocoanut Oil. H. S. Walker. (*Philippine Journ. Science*, 1906, i., 117-142.)—It is commonly accepted that cocoanut oil is peculiarly liable to turn rancid, but the author finds that when properly prepared it keeps as well as other vegetable fats and oils. It is true that the commercial product rapidly deteriorates, but this is largely due to the initial acidity developed before the expression of the oil from the coprah. The factors that cause the fat to undergo hydrolysis are the presence of water, which may be present to the amount of from 9 to 17 per cent. and a moderately high constant temperature, which allows moulds (chiefly *Aspergilli*), and certain bacteria to act upon it. A sample of coprah containing 4·76 per cent. of moisture remained free from moulds, and there was no increase in the acidity of the fat. On the other hand, coprah with 23 to 50 per cent. of moisture was found to contain several species of bacteria, but these had hardly any action upon the fat, and there were no moulds. Mould does not grow readily on pure cocoanut oil, but under certain conditions will do so, given a certain proportion of nutrient substance and of moisture. Thus, a sample of fat mixed with 1 per cent. of water and 1 per cent. of "latic"* inoculated with mould, and left in an incubator for a week, increased in acidity from 0·1 to 8·63 per cent. The part played by the action of air and light is insignificant as compared with the effect of mould action. The remedy is to dry the coprah so that it shall not contain more than 5 per cent. of moisture, and to express the oil as soon as possible afterwards. C. A. M.

Philippine Wood Oils. A. M. Clover. (*Philippine Journ. Science*, 1906, i., 191-202.)—These products are fluid resins with slight drying properties, and contain as much as 75 per cent. of oily volatile matter consisting of sesquiterpenes. They resemble copaiba and gurjun balsams in general characteristics.

Oil of Supa.—This is a mobile yellow oil obtained from the tree *Sindora Wallichii.* The author's specimen had a specific gravity of 0·9202 at $\frac{30^\circ}{30^\circ}$ C., and an optical rotation of $-31{\cdot}3^\circ$ in a 100 mm. tube at 30° C. When cooled below 20° C., it yielded a deposit of a solid hydrocarbon melting at 63° to 64° C. The oil distilled over between 143° and 149° C. under a pressure of 40 mm., leaving a fluid residue, from which the solid hydrocarbon could be separated by the addition of alcohol. A large proportion of the distillate consisted of cadinene, but no other crystalline product could be isolated. The resin had slow drying properties, and formed a hard film after exposure to the air for some weeks.

Balao: Oil of Apitong.—This is obtained from the apitong-tree, and is in common use as a varnish throughout the Philippines. As freshly obtained, it is a viscous fluid with a large amount of solid matter in suspension. It hardens on treatment with steam, but can be distilled, apparently without decomposition, over a naked flame, leaving a brittle residue. The distillate boils at 151° to 154° C. at 40 mm., and has an optical rotation of $+78{\cdot}5^\circ$ at 30° C., and specific gravity of 0·9127 at $\frac{30^\circ}{30^\circ}$ C. The author concludes that the resin is undoubtedly a sesquiterpene or a mixture of sesquiterpenes.

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[*] An albuminous liquid obtained in the native method of expressing the seeds.

Malapaho : Oil of Panao.—This is collected from *Dipterocarpus vernicifluus* in a cavity cut in the body of the tree. It dries more slowly than balao, and is thus not so extensively used. It also differs from the latter in becoming more mobile at 100° C. The fresh resin is white and viscous, but absorbs oxygen from the air and turns brown. A specimen examined by the author yielded 25 per cent. of water, 35 per cent. of oil, and 40 per cent. of solid residue when distilled over a naked flame. The sesquiterpene distilled almost completely between 256° and 261° C. at 760 mm., and after redistillation had a specific gravity of 0·9165 at $\frac{30^\circ}{30^\circ}$ C., and a rotation of − 5·4° in a 100 mm. tube.

Mayapis Resin, from the mayapis-tree (*Dipterocarpus anisoptera vidaliana*), was regarded by Tavera as identical with gurjun balsam, but the author finds that its properties are quite different. The specimen examined by him contained 15 per cent. of water, and 25 per cent. of sesquiterpene oil, which, when purified by redistillation under reduced pressure, boiled at 132° to 140° C. (17 mm.), and had a specific gravity of 0·9056 at $\frac{30^\circ}{30^\circ}$ C.

The original resin dried more rapidly than either balao or malapalio, forming a solid white film. C. A. M.

The Determination of Fatty Acids in Fats and Soaps. K. Braun. (*Seifenfabrikant*, 1906, xxvi., 127 ; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 69.)—From 1 gram to 1·5 grams of the filtered fat is saponified with alcoholic potassium hydroxide, the alcohol evaporated, the soap dissolved in hot water, and the solution exactly neutralized with hydrochloric acid, and treated with calcium chloride solution. The flask is heated on the water-bath to a temperature not exceeding 50° C., its contents then allowed to cool, and the insoluble soaps transferred to a weighed filter, on which they are washed and then dried at 100° C. They are then ignited in a weighed crucible, and the residue moistened with a few drops of nitric acid, and again ignited until constant in weight. The calcium oxide is calculated into calcium, and the difference between the result and 100 taken as fatty acids minus hydrogen. Finally, the ash is dissolved on the water-bath in 15 to 20 c.c. of N-hydrochloric acid, the solution titrated back with N-alkali, the amount of acid consumed by the ash calculated into hydrogen, and the result added to the amount taken as fatty acids. C. A. M.

On the Analysis of Soaps. W. Fahrion. (*Zeits. angew. Chem.*, 1906, xix., 385.)—The author determines water in soap by heating 2 to 4 grams of the soap with three times its weight of oleic acid (commercial olein) in an open platinum crucible over a small flame until the soap dissolves to a clear liquid. With a little practice, the point at which all the water has been driven off can be easily recognised. A determination can be completed in fifteen minutes, and the results are stated to be accurate to within 0·5 per cent. The oleic acid used should previously have been heated to 120° C. for some time. A number of the usual methods for soap analysis are also discussed in the paper. A. G. L.

Production of Methane as a Source of Error in Nitrogen Determinations by the Absolute Method. P. Haas. (*Proc. Chem. Soc.*, 1906, xxii., 81.)—The estimation of nitrogen in several organic bases by the absolute method is found to be vitiated by the presence of methane in the gas collected in the nitrometer over caustic potash. The elimination of this gas may be prevented by replacing the copper oxide ordinarily used by lead chromate, which oxidizes any methane formed.

W. H. S.

A New Method for the Quantitative Determination of Halogens in Organic Compounds. W. Vaubel and O. Scheuer. (*Chem. Zeit.*, 1906, xxx., 167.)—The method consists essentially in decomposing the organic substance with sulphuric acid, as in Kjeldahl's method, passing the evolved gases, which contain the halogens either in the free state or as hydrogen compounds, through silver nitrate solution, and weighing the haloid silver salt formed. For the decomposition, a 200 c.c. fractionating flask is used, the side-tube of which is bent upwards for a considerable length so as to act as a reflux condenser. It may also be furnished with one or two bulbs, or actually fitted to a reflux condenser if very volatile substances are to be analysed. In the neck of the flask a tap-funnel reaching to the bottom of the flask is placed, either by means of a cork or, better, by a ground-glass stopper. From 0·2 to 0·5 gram of the substance and 30 to 50 c.c. of concentrated sulphuric acid are taken. Some filter-paper or metallic copper may also be added if the halogen tends to come over in the free state. The flask is heated, at first very gently, until the substance is decomposed, a slow current of air being sucked through the flask towards the end of the operation. As receivers two Volhard's flasks are used, from which the precipitated silver salt, containing silver sulphite, is transferred to a beaker. It is then heated, at first gently, with 50 c.c. of concentrated nitric acid until the sulphite is destroyed, when the liquid is diluted so as to dissolve silver sulphate, and the haloid silver salt filtered off and treated as usual.

The test results obtained with this method on a number of bromine and iodine compounds are very good.

A. G. L.

INORGANIC ANALYSIS.

On the Reactions and Methods of Determination of Arsine.—II. Hans Reckleben and Georg Lockemann. (*Zeits. angew. Chem.*, 1906, xix., 275; *cf. ibid.*, 1905, xviii., 491.)—The authors find that the determination of arsine in a mixture with other gases (especially with hydrogen), by allowing the gases to act on silver nitrate, and either weighing the precipitated silver or else determining the arsenious oxide in the solution, does not always give good results, the values found being generally too low. On the other hand, exact results are obtained by simply noting the diminution in the volume of the gases after shaking with solutions of silver nitrate, iodine, and potassium iodide, iodic acid, or hypochlorite. The last especially gives good results in a very short time. For the qualitative detection of arsine, the darkening obtained with a fairly concentrated ammoniacal silver nitrate solution is most suitable, as it is given by mere traces of the gas. It must not be forgotten, however, that stibine, hydrogen sulphide, and phosphine also give the same reaction. A. G. L.

On the Determination of Phosphorus in Iron and Steel. L. Fricke. (*Stahl u. Eisen*, 1906, vol. xxvi., p. 276; through *Chem. Zeit. Rep.*, 1906, xxx., 82.)—One gram of steel or 0·1 gram of pig-iron (containing 0·001 to 0·003 gram of phosphorus) is dissolved in 1·2 nitric acid. To the boiling liquid 15 c.c. of a 2 per cent. solution of potassium permanganate are added, and the whole boiled for ten minutes, when 25 c.c. of a 20 per cent. solution of ammonium chloride are added to dissolve manganese peroxide, and the whole evaporated down to 30 or 40 c.c. After neutralizing with ammonia, 50 c.c. of ammonium molybdate solution are added. The precipitate is filtered off and washed free from iron with cold water; it then has the composition $(NH_4)_3PO_4.12MoO_3$. It is decomposed with an excess of standard sodium hydroxide in a flask, and the excess titrated with sulphuric acid and phenolphthalein ($2P = 46NaOH = 23H_2SO_4$). A. G. L.

Volumetric Estimation of Free Acid in Presence of Iron Salts. C. A. Ahlum. (*Proc. Chem. Soc.*, 1906, xxii., 63.)—The author overcomes the difficulty due to the inapplicability of indicators for the titration of free acid in the presence of iron salts by precipitating the iron from solution by means of sodium dihydrogen phosphate, and titrating the filtrate with standard sodium hydroxide. In the precipitation of the iron an amount of acidity is liberated directly proportional to the ferric iron present. Thus:

$$Fe_2(SO_4)_3 + 2NaH_2PO_4 = 2FePO_4 + Na_2SO_4 + 2H_2SO_4.$$
$$Fe_2Cl_6 + 2NaH_2PO_4 = 2FePO_4 + 2NaCl + 4HCl.$$

The ferric iron must therefore be estimated prior to the titration to enable its equivalent acidity to be deducted from the total found. Calcium, magnesium, and ferrous salts do not interfere with the determination, which is suitable for the analysis of natural waters containing iron salts and free acid. W. H. S.

Determination of Sulphuric Acid in Drinking-water. F. Raschig. (*Zeits. angew. Chem.*, 1906, xix., 334.)—To from 500 c.c. to 5,000 c.c. of the water, according to its content of SO_3, one-twentieth of its volume of benzidine solution (40 grams per litre) is added, and the whole allowed to stand for fifteen minutes. If no precipitate appears after this time, the water contains less than 1·5 mgms. per litre (0·105 grain per gallon) of SO_3. Any precipitate obtained is filtered off, washed very slightly, and titrated with $\frac{N}{10}$ sodium hydroxide solution. The result obtained is corrected for the solubility of benzidine sulphate in benzidine solutions by adding 1·5 mgms. per litre of SO_3. The filtrates obtained may be used for the determination of sulphur in pyrites. Any ferric iron present must be reduced with a little hydroxylamine hydrochloride before adding the benzidine solution. A. G. L.

Determination of Sulphur in Pyrites. F. Raschig. (*Zeits. angew. Chem.*, 1906, xix., 331.)—The author maintains the superiority of the benzidine over the barium chloride method for the determination of sulphur in pyrites, as the error by the former method does not exceed 0·2 per cent., whilst with the latter it may reach 0·5 per cent. The method is carried out by heating 0·8 gram of the finely-powdered ore with 5 c.c. of fuming nitric acid in a 200 c.c. flask for about thirty minutes on the water-bath; 30 c.c. of water are then added, the whole is warmed until any separated iron salt is dissolved, and then made up to 100 c.c. in a 100 c.c. flask. To 20 c.c. of this liquid 10 c.c. of a 1 per cent. solution of hydroxylamine hydrochloride are added to reduce the ferric salt, and then 500 c.c. of a dilute solution of benzidine.* The precipitate obtained is filtered after about fifteen minutes' standing through a double filter-paper placed on a porcelain disc, using suction. The precipitate should be brought on to the filter with the last portions of the solution; any adhering particles are washed on with 10 or 20 c.c. of water, whilst the funnel is still nearly full of the solution. The precipitate is then washed twice with 5 to 10 c.c. of water at a time, care being taken that it is never sucked quite dry, which would cause it to coagulate and render the subsequent treatment more difficult. The filter-papers and precipitate are then transferred to a 250 c.c. flask, using not more than 25 c.c. of water to clean the funnel. The flask is closed by a rubber stopper and shaken violently for one-half minute to break up the precipitate and filter-papers. About the calculated quantity of $\frac{N}{10}$ sodium hydroxide solution and about 2 c.c. of phenolphthalein solution are then added, when the liquid should become pink; it is heated until the colour disappears, when more alkali is added, and this treatment repeated until the pink colour is permanent even on boiling. This colour should be destroyed by at most 2 drops of $\frac{N}{10}$ hydrochloric acid.

According to the author, the method gives very exact results provided only that proper care is exercised. A determination can be completed in one and a half hours, and duplicates are easily carried out. A. G. L.

The Determination of Traces of Carbon Monoxide. R. Nowicki. (*Oesterr. Zeit. Berg. u. Hüttenw.*, 1906, i.; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 67.)—The methods most suitable for mining purposes are those based upon the oxidation of the carbon monoxide by iodic anhydride, and of these the acidimetric method is the one most generally employed. The accuracy of iodometric methods is greater than that of the acidimetric method, since it is possible to detect by their means as little as 0·002 per cent. of carbon monoxide. Colorimetric methods are mainly used for qualitative and quantitative determinations, in cases where the palladium reaction is not given, and will estimate 0·005 to 0·001 per cent. of carbon monoxide with sufficient accuracy. Preference is to be given to the benzene method, on account of the want of stability of the starch solution. The colorimetric methods can also be used for the determination of larger amounts of carbon monoxide, but there must be a suitable dilution, which reduces the accuracy of the results. C. A. M.

Magnesium Permanganate as an Oxidizing Agent. A. Michael and W. W. Garner. (*Amer. Chem. Journ.*, 1906, xxxv., 267.)—Magnesium permanganate dissolved in glacial acetic acid is an extremely powerful oxidizing agent, benzene and its homologues, and open chain compounds, such as hexane, being immediately oxidized by it to carbon dioxide and water. Pyridine is the only other solvent of a large number examined sufficiently stable towards the salt to be of any practical use for oxidation purposes, and a solution therein is only very weak, pyridine apparently exerting an inhibitory influence on the oxidizing action of the permanganate, though there is no evidence of any chemical union between them. It is considered probable that the aqueous solution of magnesium permanganate will also prove of use in cases of difficultly oxidizable substances, or where it is desirable to prevent the formation of an alkaline solution. W. H. S.

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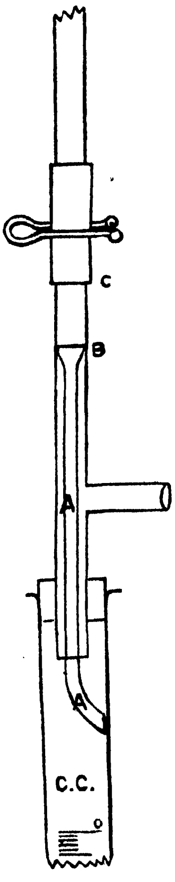
* This solution appears to contain 2 grams of benzidine per litre.—A. G. L.

APPARATUS.

Electrically-heated Muffle Furnace. G. K. (*Oesterr. Zts. Berg.-u. Hüttenw.*, 1906, vol. liv., p. 101; through *Chem. Zeit. Rep.*, 1906, xxx., 82.)—A muffle furnace due to Heraeus is described, in which the resistance material consists of platinum foil. An external resistance enables the temperature to be regulated from 700° to 1,200° C., which temperature is attained in one hour. A. G. L.



A Burette-Filling Device. Edward French. (*Chem. News*, 1906, vol. 93, p. 71.)—The apparatus shown in the figure consists essentially of a T tube connecting the end of a syphon tube leading from the stock-bottle to the burette. As the liquid enters the burette through the narrow inner tube of the vertical tube, air from the burette escapes through the annular space between the two tubes into the horizontal tube, which last may be plugged when the burette is not in use. A. G. L.

The Properties of Apparatus made of Magnesia. Kurt Arndt. (*Chem. Zeit.*, 1906, xxx., 211.)—The author has examined some apparatus, consisting of rings, tubes, tiles, crucibles, etc., made of magnesia, and supplied partly by Gebr. Siemens and Co., of Charlottenburg, and partly by the Royal Berlin Porcelain Factory. Two magnesia articles from the first firm had a porosity of 17 and 20 per cent., water entering readily. The colours were gray and yellow. The linear shrinkage on heating to the melting-point was 8 and 10 per cent. They were attacked with difficulty by acids, and contained the following percentages respectively: SiO_2,1·36 and 1·27; Fe_2O_3, 0·66 and 0·35; Al_2O_3, 0·57 and 1·30; CaO, 0·50 and 0·50 per cent. On melting, the magnesia became crystalline.

The articles furnished by the Royal Berlin Porcelain Factory were faintly yellow in colour. The porosity amounted to from 38 to 39 per cent., but the pores were so fine that water did not visibly expel air from them. They were fairly readily soluble in hydrochloric acid, and contained the following percentages respectively: SiO_2, 0·07 and 0·08; Fe_2O_3, 0·08 and trace; Al_2O_3, 0·22 and 0·46 per cent.; CaO, nil in both cases. The shrinkage up to the melting-point was 7 per cent. The melting-point was very high, above the temperature of the oxyhydrogen blow-pipe flame. This latter is contrary to the statements met with in books, which refer to magnesia containing silica, lime, and iron oxide. The new vessels must therefore not be allowed to come in contact with these bodies. Before melting the magnesia becomes crystalline, in which condition its specific gravity is 3·20 to 3·23. The specific gravity of the original material was 3·35 to 3·36. Siemens magnesia gave the values 3·30 for the original and 3·56 for the altered magnesia. The pure magnesia is not sensibly attacked during prolonged fusion with melted alkalies, although it becomes brittle on cooling. It appears to be well suited to serve as a diaphragm in the electrolysis of melted salts, which rapidly act on and destroy porcelain. A. G. L.

On Vessels for Keeping and Transporting Samples of Gas. R. Nowicki. (*Oesterr. Zeit. Berg. u. Hüttenw.*, 1906, liv., 62; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 67, 68.)—Cylindrical zinc vessels closed at each end with rubber stoppers are frequently used for the collection of mine gases, but are open to the objection that the carbon dioxide and oxygen may act upon them. Cylindrical glass vessels with capillary stop-cocks are also in common use; but it is not easy to make a direct titration of the carbon dioxide when they are used. The author also regards Hesse's flask as impracticable, and he recommends the use of screw-stoppered sample flasks, with a capacity of 500 c.c. for carbon dioxide and 1,000 c.c. for oxygen, hydrocarbons and carbon monoxide. The flasks are to be filled with water, which is allowed to run out at the place where the samples are to be taken and then closed. In making a determination of the carbon dioxide, the flask is rapidly opened, a measured quantity of standard baryta solution introduced, the flask closed and shaken, and the liquid titrated back with standard oxalic acid solution. For the determination of the gases in the other flask, the stopper is replaced under water by a T-tube, each arm of which is closed by a glass stopper or rubber tube and clip. The two tubes do not communicate with one another, but are kept distinct through the centre-piece of the T-tube, so that one serves for the introduction of water and the other for conveying the gas to the measuring apparatus. The tightness of the flask is tested by introducing 1 to 2 grams of sodium carbonate and a little hydrochloric acid, inserting the stopper, and immersing the flask in water.

C. A. M.

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REVIEWS.

CONVERSATIONS IN CHEMISTRY: FIRST STEPS IN CHEMISTRY. By W. OSTWALD. Translated by ELIZABETH C. RAMSAY and STUART K. TURNBULL. (London: Chapman and Hall. Price—vol. i., 6s. 6d. net; vol. ii., 8s. 6d. net.)

THE name of Ostwald is so generally found associated with the most recondite problems of chemical science that one almost pauses in astonishment to find a book, written by him specially for beginners, in which may be found explanations of the most simple problems and phenomena of the science, couched in the simplest and most beautiful language, but expressed with a clearness and lucidity which at once betray the master mind. The author was induced to undertake this work by a request from a firm of publishers to write a modern version of Stöckhardt's "Schule der Chemie," a work from which he, in the earliest part of his career, had received much assistance. Part I. is translated by Elizabeth C. Ramsay, Part II. by Stuart K. Turnbull. Both translators have done their work well, all traces of foreign idiom being absent. The printing, paper, binding, etc., of the book are excellent. As a guide to students, it cannot fail to be most helpful. W. J. S.

THE POLARISCOPE. By GEORGE W. ROLFE, A.M. (London: Macmillan and Co. Price 8s. net.)

The title of the book may be objected to on two grounds: First, the work treats almost exclusively of the polarimeter, which is essentially an instrument of measurement, whereas the polariscope is simply a piece of apparatus for exhibiting certain optical phenomena; secondly, the work treats of considerably more than polarimetry, for it describes chemical and other methods of testing the products, either whilst in the state of manufacture or when finished, of the various sugar industries. It is, in fact, a handbook of the scientific control of these manufactures. Written by one of long experience as technical chemist in the sugar-houses of the West Indies, the book is admirably adapted to the purpose for which it is intended, the information it conveys being invariably sound and practical. It contains numerous illustrations, useful tables, and is provided with a good index. To those engaged in this kind of work the book cannot fail to prove extremely useful. W. J. S.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST OF THE APRIL EXAMINATIONS.

OF seven candidates who entered for the Intermediate Examination, six passed: C. T. Bennett, B.Sc. (Lond.), S. H. Groenewoud, B. R. James, G. R. W. Lawson, E. H. Merritt, B.Sc. (Lond.), and C. A. Wonham, B.Sc. (Lond.). In the Final Examination for the Associateship (A.I.C.), of four examined in the branch of Mineral Chemistry, three passed: G. N. Bacon, Assoc.R.C Sc. (Lond.), B.Sc. (Lond.), F. M. G. Johnson, M.Sc. (Montreal), and E. J. Wilson, B.A. (Cantab.); of four in the branch of Metallurgical Chemistry, one passed: H. J. Bailey. Two candidates presented themselves in Organic Chemistry, and both passed: L. H. Berry and O. W. Stickland, B.Sc. (Lond.); and of seven who entered in the branch of the Analysis of Food and Drugs and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following five passed: B. J. Eaton, W. R. Mummery, F. L. Okell, R. P. Page, and R. G. Pelly. The examiners in Chemistry were Mr. W. W. Fisher, M.A., F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. The examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., F.R.S., F.I.C.

JUNE, 1906. Vol. XXXI., No. 363.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, May 2, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. H. G. Harrison, M.A., E. Miller, and F. W. Passmore, Ph.D., were read for the second time; and certificates in favour of Messrs. William Dickson, Branksome, Bridge of Weir, N.B., analytical chemist, and Gordon Wickham Monier-Williams, B.A. (Oxon), Ph.D. (Freiburg), The Lammas, Esher, Surrey, analytical chemist, were read for the first time.

Messrs. F. W. M. Ross and F. E. Thompson were elected members of the Society.

The President announced that the Anniversary Dinner of the Society would take place at the Trocadero Restaurant on Wednesday, June 13, and that, for the convenience of country members, the monthly meeting would be held on the following evening—namely, Thursday, June 14.

The following papers were read: "The Estimation of Fat in Homogenized Milk," by H. Droop Richmond; "Milk Analysis," by H. Droop Richmond and E. H. Miller; "Note on the Composition of Saffron," by Albert E. Parkes; "On the Polenske Method for the Detection of Cocoanut Oil in Butter," by S. Rideal, D.Sc., and H. G. Harrison, M.A.; and "On the Presence and Detection of Cyanogen in Java and other Beans," by R. R. Tatlock and R. T. Thomson.

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THE DETECTION OF COCOANUT OIL IN BUTTER.

By Arthur W. Thorp.

(*Read at the Meeting, March* 7, 1906.)

The addition of cocoanut stearin to butter has been an increasing practice since an odourless neutral fat was put on the market about 1897. From this date the number of samples examined showing an excessive amount of insoluble volatile fatty acids has gradually increased.

I have felt the need of a method of estimation of the insoluble acids which would be supplementary to the Reichert-Wollny determination, and I venture to think that a description of the method I have found to work satisfactorily in a commercial laboratory may be of interest.

The methods of Polenske (Analyst, 1904, p. 154) and of Muntz and Coudon

(Analyst, 1905, p. 155) involve the use of special distillation apparatus, and have not the advantage of the method I have practised. I determine the Reichert-Wollny figure, and then proceed to an approximate estimation of the insoluble volatile acids if the indications are suspicious.

I proceed as in the Reichert-Wollny test, but prefer heating the flask over a sand-bath instead of a naked flame. I distil off 110 c.c., and place the receiver in cold water for a short time. If there is any considerable quantity of cocoanut oil in the sample, oily drops will appear on the top of the distillate. I filter and titrate in the usual way, add 110 c.c. water to the flask, and distil off another 110 c.c. as before. I cool the second distillate, pour it over the same filter, and titrate the second filtrate; wash the apparatus with 50 c.c. alcohol and pour this over the filter, then wash the filter with another 50 c.c. of alcohol (90 per cent.), slightly warmed so as to hasten solution of the insoluble volatile acids; finally titrate the alcoholic solution with $\frac{N}{10}$ caustic soda and phenolphthalein.

The accompanying table of English butters blended by myself with cocoanut oil in varying proportions shows the result of my experiments. I also add the result of experiments with various foreign butters of undoubted purity and known origin.

It will be seen that the number of c.c. $\frac{N}{10}$ NaOH for pure butter was in no case higher than 8·4, whereas cocoanut oil requires 34 c.c.

	Reichert-Wollny Figure.	No. of c.c. $\frac{N}{10}$ NaOH registered by Second Filtrate.	No. of c.c. $\frac{N}{10}$ NaOH registered by Alcoholic Solution.
Butter fat ...	29·2	3·1	7·6
Cocoanut oil ...	8·0	4·0	34·0
Mixtures :			
90 per cent. butter fat, 10 per cent. cocoanut oil	27·1	3·7	10·2
80 20	24·6	4·0	15·5
70 30	23·4	4·0	18·1
60 40	21·5	3·9	20·1
50 50	19·4	4·2	21·8
40 60	17·6	4·4	24·4
30 70	15·1	4·2	26·0
20 80	13·3	4·4	27·1
10 90	10·6	4·4	29·1
Margarine ...	2·9	1·2	7·5
Italian butter ...	32·6	3·2	8·1
French butter...	31·1	3·6	7·4
	32·0	4·6	6·5
Australian butter	32·3	4·0	7·2
New Zealand butter	34·1	3·0	6·6
Argentine butter	31·0	3·1	6·2
Irish butter ...	27·8	2·9	5·2
Danish butter...	33·4	3·7	7·1
U.S.A. butter ...	29·0	3·1	8·4
Russian butter	26·1	3·0	7·3

DISCUSSION.

The PRESIDENT (Mr. Bevan) said that he had had a sample which showed a Reichert-Wollny figure of 31 and a figure of something over 3 for the insoluble volatile acids, but in which the globules were perfectly oily at 10° C., and he had felt quite satisfied at the time that there was some cocoanut oil present, though the Reichert-Wollny figure of 31 did not leave room for much. Mr. Hehner, however, had since told him of a case in which a sample giving exactly similar results was proved beyond doubt to be genuine butter, so that the oiliness of the insoluble volatile acids at 10° C. seemed, after all, to mean nothing. To his mind the most promising test seemed to be the amount of soda absorbed by a certain proportion of the insoluble non-volatile acids. In the case of cocoanut oil the insoluble non-volatile acids from 1 gramme required 4·7 c.c. of normal soda solution, whereas in the case of genuine butter the figure varied from 3·8 to 3·85 c.c.

Mr. BODMER said that he had met with a case in which the insoluble volatile acids, which had a titration value of about 3·1 c.c. of decinormal soda solution, remained quite fluid even at 10° C. The Reichert-Wollny figure, however, was 30·3, which seemed to leave no room for any cocoanut oil. He had been trying to make the process of Knud Jensen and Kirschner, published recently in the ANALYST (xxx. [1905], 205, 396) applicable to butter; but, although it worked very well with margarine, where the quantity of cocoanut oil was relatively large and the Reichert-Wollny figure low, in butter the smallness of the quantity of cocoanut oil present and the variations in the Reichert-Wollny figure for genuine butter threw out the formula altogether.

Dr. DYER said that the silver test mentioned by Mr. Bodmer had been tried in his laboratory for some time, and at first seemed likely to be useful; but on further experience it was found to break down. Some samples of butter believed to be pure gave quite distinct precipitates with silver nitrate.

Mr. F. J. LLOYD said he had found that in the spring pure butter fat sometimes contained relatively large quantities of liquid fats which were not present at other times of the year. In the case of one farm some years ago, with which he had to do, the fat was of so liquid a nature that when the milk was made into cheese the fat was actually squeezed out, and could not be retained by the curd.

Dr. RIDEAL said that he had made comparisons of the butter from different English counties, and had found considerable variation according to season and feeding. In some cases the figure for the insoluble volatile acids was more than 6 per cent. of the Reichert-Wollny figure, but as a rule English butters have a smaller ratio than found on the Continent by Polenske.

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THE COMPOSITION OF MILK.

By H. Droop Richmond, F.I.C.

(*Read at the Meeting, March* 7, 1906.)

Average Composition during 1905.—Of the 35,851 samples analysed in the Aylesbury Dairy Company's laboratory during 1905, 31,120 samples consisted of milk. The average composition of 14,828 samples of milk received from the farms is given in the table below:

Average Composition of Milk during 1905.

1905.	Morning Milk.				Evening Milk.				Average.			
Month.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January	1·0328	12·72	3·64	9·08	1·0325	13·09	4·02	9·07	1·0327	12·90	3·83	9·07
February	1·0329	12·63	3·56	9·07	1·0324	13·00	3·96	9·04	1·0327	12·81	3·76	9·05
March	1·0327	12·53	3·50	9·03	1·0324	12·89	3·90	8·99	1·0326	12·71	3·70	9·01
April ...	1·0326	12·41	3·43	8·98	1·0323	12·72	3·75	8·97	1·0325	12·57	3·59	8·98
May ...	1·0329	12·33	3·31	9·02	1·0324	12·76	3·77	8·99	1·0326	12·55	3·54	9·01
June ...	1·0327	12·22	3·27	8·95	1·0322	12·62	3·70	8·92	1·0324	12·42	3·49	8·93
July ...	1·0321	12·29	3·44	8·85	1·0315	12·52	3·76	8·76	1·0318	12·41	3·60	8·81
August	1·0318	12·30	3·51	8·79	1·0314	12·69	3·93	8·76	1·0316	12·50	3·72	8·78
September	1·0322	12·55	3·63	8·92	1·0319	12·92	4·02	8·90	1·0320	12·74	3 83	8·91
October	1·0327	12·72		9·04	1·0323	13·01	4·01	9·00	1·0325	12·86	3·84	9·02
November	1·0326	12·89	3·83	9·06	1·0322	13·16	4·15	9·01	1·0324	13·03	3·99	9·04
December	1·0325	12·72	3·73	8·99	1·0323	12·96	3·97	8·99	1·0324	12·84	3·85	8·99
Average	1·0325	12·53	3·54	8·99	1·0321	12·86	3·91	8·95	1·0323	12·70	3·73	8·97

The mean composition is practically the same as that found in 1904, the average percentage of fat differing only by 0·01 per cent. As usual, May and June are the lowest months for fat, and the winter months are the highest, while the usual drop in solids-not-fat was found in July and August. The difference between morning and evening milk, 0·37 per cent., agrees with that previously found.

Human Milk.—A sample of human milk was examined; the composition was:

Total solids	13·97	per cent.
Fat	5·61	,,
Sugar	6·98	,,
Proteids	1·27	,,
Ash	0·18	,,
Solids-not-fat	8·36	,,

Zeiss refractometer figure of the fat, at 35° C., 51·3°.

The fat globules were extremely large, ranging from 0·035 mm. in diameter to 0·002 mm., and the cream rose very rapidly.

Ass's Milk.—A sample of ass's milk was analysed by Mr. J. A. Goodson. It was sour when received, but had not curdled, and was still in a state which allowed the analysis to be performed. His figures were:

Specific gravity	1·0362	
Total solids	10·27	per cent.
Fat	1·45	,,
Sugar	5·65	,,
Proteids	2·09	,,
Ash	0·54	,,
Solids-not-fat	8·82	,,
Acidity as lactic acid	0·54	,,

This milk is slightly above the average in fat, proteids, and ash.

Adulterated Cream.—Among the newer forms of adulteration of cream, the mixing of foreign fat and of condensed milk may be mentioned.

A sample of the former kind of cream contained:

Total solids	35·55	per cent.
Fat	28·95	,,
Sugar	3·28	,,
Proteids	2·36	,,
Ash	0·58	,,
Solids-not-fat	6·60	,,

The fat yielded the following figures:

Reichert-Wollny figure	21·5 and 21·8 c.c.
Potash absorption	21·63 per cent.

A cream adulterated with condensed milk had the following composition:

Total solids	48·51	per cent.
Fat	38·25	,,
Ash	0·76	,,
Solids-not-fat	10·26	,,

A recently-introduced Adulterant for Butter.—Butter is now adulterated with casein. A sample of the preparation used was examined. This sample was not exactly in the form in which it is added to butter, as the exact quantity of sodium carbonate necessary for neutralization and a considerable amount of salt are added before it is worked in. The composition was:

Water	65·47	per cent.
Fat	0·08	,,
Sugar	2·24	,,
Soluble proteids	0·69	,,
Ash of soluble portion	0·35	,,
Ash of insoluble portion	0·67	,,
Casein (by difference)	30·50	,,

From the large amount of chlorides present, this appears to be a badly-washed casein which has been precipitated by hydrochloric acid from separated milk.

To see the change of composition of butter, 5 per cent. of this was worked into some butter. The salt, which was added to the genuine butter, was omitted in the portion to which the casein was added. The composition of the two samples was :

	Genuine Butter.	Butter plus Casein.
Water	13·51	16·02
Fat	83·92	82·08
Curd	0·96	1·90
Casein	0·23	1·08
Salt	1·61	None

The casein was determined by extracting the "curd" with dilute ammonia, filtering, and precipitating with dilute acetic acid. I have found the average amount of casein in genuine butters (unwashed) to be 0·38 per cent., and not to exceed 0·50 per cent.

A New Preservative.—A sample of preservative which came into my hands proved to be acid potassium fluoride (KHF_2); it is in the form of semi-transparent flat crystals, very little hygroscopic, and very soluble in water. I found:

Potassium	49·7	per cent., calculated	50·1	per cent.
Acidity as HF	24·9	,, ,,	25·5	,,
Loss on ignition	25·45	,, ,,	25·5	,,

DISCUSSION.

The **PRESIDENT** (Mr. Bevan) said that he was afraid that the adulteration of butter with casein was only too common. He had lately met with several samples containing 4 to 5 per cent. of curd, always accompanied by a large proportion of water. Milk-sugar also was generally present, pointing to the addition of condensed milk.

Mr. **BLOUNT** said that he should be interested to hear whether the use of potassium hydrogen fluoride was at all common, because some fifteen years ago, when fluorides were suggested as a preservative for fish, he had himself for the purpose of experiment taken a small quantity of sodium fluoride, and had in consequence suffered from acute indigestion.

Mr. **RICHMOND** said that he did not know that potassium hydrogen fluoride was much used. It came into his hands in rather a roundabout way from the United States. It was said to be perfectly harmless and to preserve all kinds of food, and was put on the market under the name of "acid potassium oxalate!"

Mr. L. **MYDDELTON NASH** asked in what way the extra casein was worked into the butter; whether it was churned in in a freshly precipitated condition, or was first dissolved in a trace of alkali. He had lately received a formula from America, one of the constituents of which was "potassium oxalate." He had been at a loss to understand the reason for including such an ingredient, but Mr. Richmond's statement that potassium fluoride was sold as such somewhat enlightened him.

Mr. A. E. **PARKES** said that in 622 samples of milk which he had examined during the past year under the Sale of Food and Drugs Acts, the average proportion of fat was 3·52 per cent., even including 21 per cent. that were adulterated, showing how low the standard for fat adopted by the Board of Agriculture is.

Mr. F. J. Lloyd thought that erroneous ideas were likely to be derived from the averages of such very large numbers of samples as were dealt with in this paper. In his experience the milk produced during June had never been so poor in fat as it was in 1905. At the Bath and West of England Society's Show in particular a much larger percentage of the cows than usual were disqualified for the milking trials because their milk was below the required standard of 3 per cent. With regard to cream, he had been informed privately that a large quantity containing foreign fat was now being put on the market. He had recently found fluorides in both butter and cream.

Mr. J. F. Liverseege said that he had lately met with cream containing starch. It was not very easy to estimate the proportion. He had found the simplest way to be to determine the total solids, fat, milk-sugar, ash, and nitrogen, and to take the starch by difference. The average percentage of fat in the samples of milk bought under the Act in Birmingham in 1905 was 3·6. He should like to ask Mr. Richmond how far the cows represented by his figures were stripped; and he should also be interested to hear Mr. Richmond's opinion as to the effect of different modes of feeding on the quality of the milk.

Dr. Voelcker said that it was certainly the case that both at the Bath and West of England and at the Royal Agricultural Society's Shows in 1905 there was a larger number than usual of morning milk samples showing low results; but he was far from thinking that such samples were really to be taken as fair guides. Considering all the circumstances attending the exhibition of animals away from their own homes, and amid the disturbing surroundings of agricultural shows, it was only a very well-trained animal that would be likely to give good results. Indeed, he would go so far as to say that what show animals were required to do was something abnormal—namely, to produce a larger quantity of milk than they would produce under normal conditions, the prizes, rightly or wrongly, being generally given for the largest quantity of milk, provided that it reached a certain standard of quality. He thought that conclusions drawn from the results of such show-yard tests had little bearing on the quality of the average milk supplied by the farming industry of this country to the public.

Mr. M. Wynter Blyth thought that Mr. Richmond's figures could not be considered to represent quite normal milk, because the farms were specially selected, and the farmers, knowing that the milk would be analysed, would take care to keep up to a certain standard. At Brighton samples of milk were regularly taken at railway-stations as they came direct from the farmers, and last year the proportion of these falling below the standard had been greater than ever before. That might be attributed either to a bad year, or to efforts on the part of the farmers to "break down" the milk to the standard of 3·0 per cent., as laid down by the Sale of Milk Regulations. He thought that the Regulations had had a bad rather than a good effect, and he considered that it would be better to have a fixed standard, and to allow no excuse whatever for departing from it. Mr. Richmond's figures seemed to support that view, for although in his (the speaker's) opinion they could not be regarded as normal for the whole country, they showed what could be done on a well-managed farm.

Mr. Richmond said that the casein was precipitated from skim milk with acid and pressed fairly dry; sufficient sodium carbonate was added to neutralize the acid exactly, and the casein was then worked into the butter. There were, practically speaking, no samples low in solids-not-fat, and it was only in May and June that any extensive numbers of samples low in fat were observed. There also were a few samples low in fat in April and July, but in the other months of the year they were rare. It was only the morning milk which need be considered, as even in May and June the fat in the evening milk hardly ever fell below 3·0 per cent. The table below gives the percentage of samples of *morning milk* falling below 3·0 per cent. of fat during May and June for each year since 1900:

MORNING MILK ONLY.

Month.	Fat 2·9 to 3·0.	Fat 2·8 to 2·9.	Fat 2·7 to 2·8.	Fat below 2·7.
May, 1900	4·1	0·9	0·4	—
June, 1900	3·0	2·0	0·4	0·2
May, 1901	4·0	2·0	1·6	0·4
June, 1901	2·0	1·4	—	0·4
May, 1902	2·2	2·0	0·8	—
June, 1902	4·7	1·5	0·4	—
May, 1903	2·0	0·9	—	—
June, 1903	1·7	0·6	0·8	—
May, 1904	2·7	1·8	0·9	0·5
June, 1904	3·6	3·2	0·8	0·2
May, 1905	6·0	2·0	0·6	0·2
June, 1905	3·1	2·0	0·2	—

His experience was that, practically speaking, 1905 was no worse a year for milk than any other of the last five years. A little variation occurred from year to year, sometimes in the one direction, sometimes in the other; but although the milk of 1905 was a little less rich than that of 1903, which was one of the best years, it was better than that of 1900, and about equal to that of the other years. He did not know of any very good method for the estimation of starch in cream. Probably that adopted by Mr. Liverseege would give fairly good approximate results. He thought that in most cases the cows would be thoroughly stripped, because the milk had to be supplied to a certain standard. He was afraid that the shortness of the time remaining would not allow of his going into the question of the effect of feeding. He thought the figures he had given were fairly representative of normal milk, although the farms were to some extent selected, because the selection had no reference to the composition of the milk, but to the water-supply and sanitation, and in his experience the farmers did not take any special steps to obtain milk of more than average quality because they knew that the milk was regularly analysed, as each farmer believed that his milk was richer than anyone else's.

Dr. Schidrowitz suggested that the starch might be estimated simply by extracting the cream with water, taking out the fat with ether, treating the residue with a solution of malt extract after gelatinizing, filtering off, and determining the optical rotation and copper-reducing power.

THE DETERMINATION OF HIGHER ALCOHOLS IN SPIRITS.—II.

By PHILIP SCHIDROWITZ, Ph.D., and FREDERICK KAYE, A.R.C.Sc.

In our first paper on this subject (Analyst, June, 1905) we gave the details of our revision experiments on the Beckmann process, and also some few facts concerning the Allen-Marquardt process. In the present communication we wish to record a series of experiments on the Allen-Marquardt process and on the colorimetric (sulphuric acid) method.

B. The Allen-Marquardt Process.

The purification of materials and the apparatus employed were described in our previous paper, and need not, therefore, be referred to again here.

The first series of figures (Table I.) relates to the oxidation of amylic alcohol dissolved in carbon-tetrachloride with chromic acid mixture—that is to say, the quantities of the higher alcohol stated to be present were added directly to the carbon-tetrachloride, and were not obtained by extraction from a dilute alcoholic solution of brine. The object was to ascertain the behaviour of amylic alcohol as such in varying proportions and conditions of the process of oxidation.

Table I.

Oxidation of Amylic Alcohol in Carbon-tetrachloride Solution.

(Carbon-tetrachloride 100 c.c.; oxidation mixture, 5 grams bichromate of potash, 30 c.c. H_2O, 1·5 c.c. strong H_2SO_4.)

No. of Experiment.	Weight of Amylic Alcohol present (Grams).	Weight of Amylic Alcohol found (Grams).	Percentage Gain or Loss.	Duration of Oxidation (Hours).	Remarks.
1	0·0990	0·0952	− 3·8	8	
2	0·1000	0·0942	− 5·8	8	
3	0·1066	0·0982	− 6·0	8	
4	0·1178	0·1007	−14·5	8	Double quantity of oxidation mixture.
5	0·1640	0·1610	− 1·8	8	
6	0·1740	0·1760	+ 1·1	8	
7	0·1770	0·1810	+ 2·0	8	
8	0·1874	0·1582	−15·5	8	
9	0·2367	0·2112	−15·0	8	
10	0·3324	0·2877	−13·4	8	Double quantity of oxidation mixture.
11	0·1864	0·1703	− 8·0	10	
12	0·1970	0·2020	+ 7·0	10	
13	0·4140	0·3362	−18·7	8	
14	1·5338	1·1325	−26·1	8	Triple quantity of oxidation mixture.

From the above table the following conclusions may be drawn: When the quantity of higher alcohol is not in excess of that ordinarily found by the process in commercial spirits—*i.e.*, up to 0·15 per cent.—the results are very accurate as far as the oxidation is concerned, with a boiling period of eight hours. When the quantity of higher alcohol is substantially above 0·15 per cent., a boiling of ten hours appears desirable (*cf.* Nos. 11 and 12 with Nos. 8, 9, and 10). When the quantity of higher alcohol is markedly over 0·3 per cent. (Nos. 13 and 14) a variation of the method is obviously necessary. This is of little practical importance, as commercial spirits such as whisky and brandy rarely contain more than 0·13 per cent.

Table II. gives the results obtained by adding known quantities of amylic alcohol to 100 c.c. of pure 50 per cent. ethylic alcohol, diluting with brine and extracting with CCl_4 in the usual manner, and then oxidizing as above.

TABLE II.

Results of the Allen-Marquardt Process obtained by Working with 50 per cent. Solutions of Ethylic Alcohol containing known Amounts of Amylic Alcohol.

No. of Experiment.	Weight of Amylic Alcohol taken (Grams).	Weight of Amylic Alcohol found (Grams).	Percentage Gain or Loss.	Duration of Oxidation (Hours).
15	0·0610	0·0616	+ 1·0	9
16	0·0942	0·0915	− 2·8	8
17	0·1004	0·1056	+ 5·0	9
18	0·1407	0·1390	− 1·2	8
19	0·1472	0·1415	− 3·9	10
20	0·1992	0·1624	− 18·4	8
21	0·1992	0·1654	− 17·1	8
22	0·2008	0·1693	− 15·6	8
23	0·2008	0·1725	− 14·0	8
24	0·2060	0·2187	+ 6·0	10
25	0·2077	0·2128	+ 2·4	10
26	0·3776	0·3243	− 14·0	10
27	0·3450	0·3432	− 0·5	12

The above results plainly indicate that the process as a whole gives very accurate results when the quantity of higher alcohol is not greater than, roughly, 0·15 per cent. (Nos. 15 to 19). When the percentage of higher alcohol substantially exceeds 0·15 per cent., but does not rise materially above 0·2 per cent., it is necessary to prolong the period of boiling somewhat (*cf.* Nos. 20 to 23 with Nos. 24 and 25). It is obvious, however, that quantities up to 0·35 per cent. can readily be dealt with if the duration of oxidation is extended to twelve hours. In view of the fact, to which we have alluded above, that commercial spirits rarely exceed 0·15 per cent., and that it has now been amply demonstrated that this amount can be successfully dealt with in eight hours with brisk boiling, we do not advocate extending the time to ten or twelve hours, but would suggest that if a result showing more than 0·14 per cent.

be obtained, the analysis should be repeated after diluting the spirit with a convenient quantity of pure 50 per cent. alcohol, or taking a smaller quantity of the sample.

We may add that we have examined several hundred samples of commercial spirits (whisky* more particularly) by this method, and only in the rarest cases have we obtained figures which might be regarded as doubtful. With a little practice the process works smoothly, and the abnormal results which may cause trouble to the novice will be found to become very exceptional indeed. When it is considered that the oxidation is carried out in an open vessel—*i.e.*, not under pressure in a sealed flask or tube—we think it will be admitted that the accuracy of the results obtainable is remarkable. We have little doubt that if the oxidation were performed in a pressure bottle the results might be still more satisfactory, and we intend to make some experiments in this direction in the future. We propose also to make some further experiments in which butylic and propylic alcohols will be substituted for amylic alcohol.

Before leaving the Allen-Marquardt process, it is necessary to refer to some observations and experiments we have made concerning the estimation and nature of the "mineral acid." Allen (ANALYST, 1891) states that the "mineral acid" formed in the course of the process corresponds to, roughly, 2 c.c. of decinormal baryta, and that the actual amount may be estimated by titrating in the first place with baryta, using methyl orange as indicator, and afterwards adding phenolphthalein in order to estimate the organic acids. In the course of a very large number of estimations we noticed that the amount of "mineral acid" thus determined varied widely, but that, almost without exception, it amounted (expressed in terms of the number of c.c. of decinormal baryta employed) to, roughly, one-tenth of the whole. In view of the singularity of this fact, we thought it desirable to make some estimations of chlorine in the solutions finally obtained, in order to ascertain whether this corresponds to the amount of "mineral acid" found by titration. The following figures were obtained:

TABLE III.

No. of Experiment.	Chlorine found (Grams).	Chlorine calculated (Grams).
1	0·0023	0·0070
2	0·0012	0·0085
3	0·0012	0·0108
4	0·0039	0·0136
5	0·0014	0·0094
6	0·0007	0·0107
7	0·0006	0·0139
8	0·0014	0·0065

It will be observed that in every instance the amount found was very much smaller than the calculated quantity. Indeed, whereas the amount calculated generally approaches to or exceeds a centigram, the amount found is so small in most cases as to be within the experimental error.

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* *Cf.* Schidrowitz and Kaye, "The Chemistry of Whisky"—II., *Journ. Soc. Chem. Ind.*, June, 1905.

It is obvious that the only mineral acid which might reasonably be supposed to be present is hydrochloric acid. These results led us to think that the so-called "mineral acid" might not be mineral acid at all, but that its apparent presence might simply be due to the fact that some organic acids are not without effect on methyl orange. It has long been known (*cf.* Glaser, "Indikatoren der Acidimetrie u. Alkalimetrie," p. 23 *et seq.*, and Schidrowitz, ANALYST, August, 1903) that acetic acid in aqueous solution reacts with methyl orange, and, on the other hand, that this reaction may be prevented, or the pink reaction colour caused to disappear by the addition of a sufficiency of a non-electrolyte such as alcohol or acetone, but we are not aware that the action of aliphatic acids on the indicator has been the subject of quantitative examination. If the explanation of the nature of the "mineral acid" which suggested itself to us were correct, it would follow: (1) That the addition of alcohol or acetone to the ultimate distillates obtained in the Allen-Marquardt process would prevent the appearance of, or, if added subsequently to the addition of the indicator, cause the disappearance of the pink coloration reputedly due to "mineral acid"; and (2) that aliphatic acids such as butyric and valeric acids would in conditions of solution approximating to those of the Allen-Marquardt process show similar results.

Ad. 1.—On examining a number of ultimate distillates of the Allen-Marquardt process on the lines indicated it was found that when an equal volume of alcohol or acetone was added to the solution prior to the addition of the indicator the colour obtained was a pure yellow, and similarly, that when the alcohol or acetone was added to the solution after the indicator had been run in, the solution, which had been a distinct pink, reverted to a plain yellow. In these same test cases also it was found (by titrating an aliquot part of the solution) that the apparent "mineral acid" was quite normal—*i.e.*, about one-tenth of the whole.

Ad. 2.—Some preliminary experiments have been made with acetic, butyric, iso-butyric, and valeric acids, and these entirely confirm the hypothesis stated above. The following figures were obtained:

TABLE IV.

Acetic Acid.

No. of Experiment.	Volume of Solution.	Acetic Acid calculated (Grams in Solution).	Acetic Acid found (Grams in Solution).	C.C. $\frac{N}{10}$ Baryta, using Methyl Orange (=apparent Mineral Acid).	C.C. $\frac{N}{10}$ Baryta, Total for Methyl-orange and Phenolphthalein Titrations.	Ratio of Total Titration to Methyl-orange Titration.
1	100 c.c.	0·1334	0·1331	3·35	22·18	6·5 : 1
2	,,	0·0667	0·0665	1·35	11·09	8·2 : 1
3	,,	0·0333	0·0332	0·68	5·54	8·2 : 1
4	,,	0·0333	0·0332	0·68	5·54	8·2 : 1
5	,,	0·0266	0·0276	0·54	4·60	8·5 : 1
6	,,	0·0200	0·0192	0·40	3·52	8·8 : 1
7	,,	0·0133	0·0146	0·27	2·44	9·0 : 1

			Butyric Acid.			
8	100 c.c.	0·1614	0·1654	2·24	18·80	8·4
9	,,	0·1077	0·1100	1·19	12·50	10·1
10		0·0538	0·0544	0·54	6·19	11·4
11		0·0215	0·0228	0·22	2·60	12·0
			*Iso-butyric Acid.**			
12	100 c.c.	0·0835	0·0882	1·64	10·02	6·1
13	200 c.c.	0·0835	0·0896	1·47	10·18	6·9
14	100 c.c.	0·0417	0·0448	0·73	5·09	6·9
15	200 c.c.	0·0417	0·0448	0·65	5·09	7·8
16	250 c.c.	0·0417	0·0448	0·39	5·09	10·0
			Valeric Acid.			
17	50 c.c.	0·0211	0·0209	0·33	2·05	6·2 : 1
18	100 c.c.	0·0422	0·0419	0·68	4·11	6·2 : 1
19	150 c.c.	0·0422	0·0419	0·57	4·11	7·2 : 1
20	200 c.c.	0·0422	0·0419	0·49	4·11	8·3 : 1
21	250 c.c.	0·0422	0·0426	0·49	4·18	8·5 : 1

From the above figures it would appear that there is some quantitative relationship between the total acid found by titration and the apparent "mineral acid" on the one hand and between the concentration of the solution and its total volume (as compared with the amount of indicator employed) on the other. The above experiments are of a preliminary nature only, and it would be premature, therefore, to discuss the action of methyl orange on aliphatic acids further at present. We intend to pursue this matter in the future, as it presents a wider interest than that which associates it with the present communication; but as far as the latter is concerned, it is quite clear that the result of the preliminary titration for "mineral acid" in the Allen-Marquardt process may be neglected when the figure obtained is less than or roughly equal to one-tenth of the whole. In doubtful cases, or where great accuracy is desired, a gravimetric estimation of chlorine is indicated.

C. The Colorimetric (Sulphuric Acid) Process.

This process, as is now fairly well known in this country (*cf.* Girard et Cuniasse, "Manuel pratique de l'Analyse des Alcools et des Spiritueux"; Paris, Masson, 1899) is based upon the fact that certain higher alcohols, when heated with strong sulphuric acid, give rise to a more or less marked coloration, the intensity of the coloration observed being taken as a measure of the quantity of the higher alcohols present. Numerical expression is given to the colour intensity by comparing the liquid under examination with a standard control solution or solutions, iso-butyric alcohol being the standard higher alcohol generally employed for this purpose. We have repeatedly (ANALYST, June, 1905; *Journal of the Society of Chemical Industry*, June, 1905, etc.) pointed out the cardinal defects which such a process must possess.

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* It is obvious that the iso-butyric acid employed was not quite pure.

In view of the fact that others besides ourselves (Girard et Cuniasse, *loc. cit.*; Mohler, *loc. cit.*; Vasey, "The Analysis of Potable Spirits"; Allen, *Journal of Federated Institute of Brewing*, 1897; Veley, *Journ. Soc. Chem. Ind.*, May 15, 1906, etc., and numerous private communications to the authors) have published facts and figures demonstrating the defects of *principle* inherent in the sulphuric acid process, we cannot but express surprise at the assurance with which results based on this process are put forward. The defects of the process may be summarized under two headings: (1) Defects of Principle, and (2) Defects in Practice.

Defects of Principle.—The main objection to the process under this heading is contained in the fact that the coloration produced by various higher alcohols differs enormously. Thus, if the coloration due to iso-butyric alcohol be taken as = 10, then the colorations due to caprylic alcohol, œnanthic alcohol, amylic alcohol, and the propylic alcohols are 11, 7, 3, and 0 respectively (Mohler, *loc. cit.*). More recently Veley (*loc. cit.*) has published some experiments which throw doubt even on the comparative accuracy of these figures, and it appears probable that the colorations produced are really not due to the higher alcohols at all, but to the traces of impurities they contain. If this is so, it goes far towards explaining the widely different results obtained by different chemists when working on identical samples. However that may be, it must be remembered that the higher alcohols in any given commercial spirit are mixtures, and there is little doubt, in view of the work of Bell, Rabuteau, Ordonneau, and others, that the proportions of the various higher alcohols in any given *type* of spirit (*i.e.*, whether pot still or patent still whisky, or brandy, etc.) differ according to the type. Moreover, in view of recent work on the formation of the higher alcohols in fermenting worts (Ehrlich), it is plain that even in the same type of spirit the composition of the "fusel" will vary considerably.

In consideration of all these facts, the argument that if a particular alcohol be chosen as the standard or "type," that all results obtained will be of a strictly comparative nature, seems to us to be absolutely untenable; for, in order that this should be the case, it would have to follow that the proportions of the various higher alcohols to one another in any given spirit would have to be a constant, and that the higher alcohol used for the control should be of the same degree of purity (or rather impurity) at all times. There is, however, another grave objection of principle to this process, and that is that there are undoubtedly other substances in spirits besides higher alcohols and besides those which are purposely removed by means of potash or some other dealdehyding reagent), which give a coloration with sulphuric acid. We are unaware of the exact nature of these substances, of their quantity, and of the intensity of coloration they produce, and from our very numerous experiments we have reason to believe that the presence of these substances is of far greater practical importance than has hitherto been suspected (*cf.* our paper on this subject, ANALYST, June, 1905, and discussion thereon). It has been suggested that even if this process does not yield results referring to higher alcohols only, it is, nevertheless, a valuable comparative index of the nature of a spirit. To this we demur, mainly because of our lack of exact knowledge concerning the substances other than higher alcohols which do produce coloration, and because our analytical experience (see tables below) has shown us that the coloration produced by these substances is out of all proportion to the actual higher alcohols, and that it is not even in the narrowest sense indicative of the nature of the spirit examined. It appears to us, therefore, in any case unwarrantable to place on the same numerical or quantitative basis figures referring, on the one hand, to substances of a definite chemical nature, such as the esters, acids, and aldehydes, and, on the other hand, to higher alcohols plus substances of unknown quantity or colorimetric effect. That is to say, assuming that the "colorimetric test" is an index figure of quality, it should be separately returned, and not included in the quantitative "coefficient of impurities."

Defects in Practice.—Of a very real nature is the difficulty of preparing specimens of "pure" iso-butylic (or amylic) alcohol of constant colorimetric intensity for purposes of the control or "type" solutions. If Veley's statements are accepted, this appears to be an absolutely insurmountable objection to the process. The remaining defects in practice (apart from those involving questions of principle) are mainly ascribable to the numerous modifications in detail practised by different workers. To begin with, no less than four different de-aldehyding reagents have been proposed. All of these are apparently in use, and it is unfortunately a fact that the results may differ very considerably, according to the reagent employed. The method of heating the alcoholic solution with the sulphuric acid, again, is of the greatest practical importance (see below), and yet no two workers appear to use exactly the same method. Finally, we are by no means sure that the same method of calculating the amount of "higher alcohols" from the observed coloration is employed by all chemists. We have come across results which have indicated to us that those responsible for the figures are under the apprehension that the coloration observed stands in direct ratio to the "higher alcohols." This, of course, is only the case when the liquid under examination and the control solution exhibit practically equal intensities. We now proceed to give some figures bearing on the points raised above.

1. *Figures illustrating Defects in Principle.*—In a recent number of the *Journal of the Society of Chemical Industry* (June, 1905) we published a series of analyses of whiskies in which the higher alcohols were determined by the Allen-Marquardt and the colorimetric (using an amyl alcohol control) methods respectively. The number of analyses was about one hundred. The results by the latter process are throughout much higher than those obtained by the former; but it would follow that if the composition of the "fusel" was substantially the same in all whiskies, and if the other substances producing coloration were negligible or present in quantities bearing some fixed ratio to the "fusel" (*i.e.*, higher alcohols), the ratio of the results obtained by the two processes would be practically a fixed quantity. This is, however, far from being the case, even for whiskies of the same class. The following table shows the maximum and minimum ratios observed in each individual class of whisky for results obtained by the two processes in individual samples of the spirit:

TABLE V.

Ratios of Figures obtained by the Allen-Marquardt and Colorimetric Processes respectively (Amylic Control).

Type of Whisky.	Ratio of Allen-Marquardt to Colorimetric Result.	
	Maximum Ratio.	Minimum Ratio.
Highland malt	6·1	1 2·0
Lowland „	5·5	1 1·9
Campbeltown	4·6	1 2·0
Islay	4·6	1 3·1
Grain (patent still) ..	6·6	1 0·8

It will be noticed that the variations in each class of whisky are very large. Since the results on which the above figures are based were published we have made a considerable number of comparative experiments, in which both the iso-butylic and amylic controls were employed. We append a few of the results obtained herewith:

TABLE VI.

Parallel Determinations of "Higher Alcohols" by Three Methods.

No. of Experiment.	Type of Spirit.	Allen-Marquardt (Oxidation Method).	Colorimetric Metho	
			*Iso-butylic Control.	Amyli
1	Patent still	300*	204*	10
2	„	70	64	102
3	„	58	120	3
4	„	52	46	
5	Pot still	145	156	6
6	„	185	284	11
7	„	147	150	4
8	„	217	208	3
9	„	351	248	768
10	Blend	146	155	447
11	„	193	212	431
12	„	92	142	354
13	„	76	76	107
14	„	77	149	191
15	„	43	115	131
16	„	81	208	245
17	„	47	181	334
18	„	53	250	410

The numbers refer to parts per 100,000 of absolute alcohol throughout.

The above results, which we have selected from a large number, are fairly representative. It will be noticed (1) that the results obtained with the iso-butylic control are generally much lower than those obtained with the amylic; (2) that in some cases the results by the oxidation and colorimetric (iso-butylic) methods approximate very closely, but that, on the other hand, they frequently diverge to a very marked extent; (3) that generally the results by the oxidation method are a good deal lower than those by the colorimetric (iso-butylic), but that this is by no means always the case; (4) that in some cases the results by all these processes are fairly close. It is obvious that the greatest divergence will occur in those cases in which the mean composition of the higher alcohols differs widely (as far as their colorimetric properties are concerned) from the alcohol used as a standard type, or when the proportion of substances other than higher alcohols, which react colorimetrically with sulphuric acid, is high. It should be noted that the above colorimetric results were obtained by heating in the water-bath for an hour with sulphuric acid, after the spirits had been de-aldehyded by means of Hewitt's reagent (phenylhydrazine *p*-sulphonate of lime).

TABLE VII.

Figures illustrative of Defects in Practice.

(*a*) *Use of Different De-aldehyding Reagents.*

No. of Experiment.*	Reagent used.	
	Hewitt's Reagent.	Meta-phenylene-diamine-hydrochloride.
1	194	184
2	182	189
3	345	227
4	150	179
5	528	480

TABLE VIII.

(*b*) *Use of Different Methods of Heating* (*Control, Iso-butylic Alcohol*).

No. of Experiment.†	Methods of Heating.	
	Naked Flame.	Water-Bath (one hour).
1	194	116
2	150	116
3	345	145
4	238	131
5	227	126

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* The control used in the above series was iso-butylic alcohol. The heating was conducted over a naked flame.

† De-aldehyding reagent used in above series—Hewitt's reagent. All the above were whiskies.

In the following series are set out the results obtained by five different methods :

Table IX.

Process.

No. of Experiment.	Allen-Marquardt.	Colorimetric (Iso-butylic Control throughout).			
		De-aldehyding Reagent.			
		Hewitt's Reagent.		M.-phenylene-diamine-hydrochloride.	
		Naked Flame.	Water-bath.	Naked Flame.	Water-bath.
1	103	194	116	184	97
2	87	150	116	179	140
3	69	160	138	154	139
4	112	345	145	227	185
5	85	238	131	244	169
6	78	258	177	274	84
7	92	227	126	191	172

All the above were whiskies.

It will be observed that the "water-bath" colorimetric results are closer to the oxidation figures than those obtained by heating over a naked flame.

De-aldehyding Reagents.—The only reagent which to our knowledge removes the aldehydes completely and without apparently bringing about any secondary reactions is phenyl-hydrazine-sulphonate of lime (Hewitt's reagent). Meta-phenylene-diamine-hydrochloride, which is strongly recommended by some French chemists, does not always remove the furfural completely from commercial spirits, as the following figures show :

Table X.

No. of Experiment.	Total Furfural.	Furfural not removed by M.-phenylene-diamine-hydrochloride.
1	2·9	0·75
2	2·8	0·78
3	3·0	0·92
4	2·5	0·46

These figures were obtained by working on whiskies. The quantity of the de-aldehyding salt employed was 1·5 grams for 50 c.c. of spirit. It appears that the failure of the reagent to remove furfural completely is due to a secondary reaction, for solutions of furfural in pure 50 per cent. alcohol, containing far more furfural than the whiskies referred to, were completely defurfuralized by the reagent. The same result was obtained with solutions of furfural *plus* acetic aldehyde in pure

50 per cent. alcohol. Thus, a 50 per cent. ethylic alcohol solution to which 10 parts of furfural per 100,000 of absolute alcohol had been added was completely defurfuralized by means of 1·5 grams of the reagent; and the same result was obtained with a solution containing 10 parts of furfural and 24·5 parts of acetic aldehyde. It is obvious that the non-removal of all the furfural in the case of commercial spirits is another source of error in the higher alcohol figures obtained by means of the diamine reagent, inasmuch as furfural, as is well known, acts as an intensifier to the action of the sulphuric acid. It is curious that, nevertheless, the results obtained with the reagent in question are generally lower than those recorded when Hewitt's reagent (which completely removes furfural and aldehydes) is used. We believe that this is due to the fact that the meta-phenylene-diamine reagent enters, in the case of commercial spirits, into secondary reactions with some of the less known "impurities." The facts recorded above indicate that this is so, but we have also noticed that a peculiar fluorescence is produced by this reagent, either in the spirit before distillation, or in the distillate, or in both. We have also observed the fact that in those cases in which furfural is incompletely removed the fluorescence is only marked in the spirit prior to distillation, and that the distillate is then colourless. On the other hand, when the phenomenon of fluorescence is slight in the liquid prior to distillation, but strongly developed in the distillate, the latter shows little or no furfural.

Regarding other de-aldehyding reagents, we have found phosphate of aniline useless, as its use invariably results in the presence of aniline in the distillate. We have made some experiments with caustic alkalies, but the number of experiments made is not sufficient to warrant our expressing any definite opinion. We can only say that if, after considering all the facts set out in this paper, some of our colleagues still desire to continue the use of a colorimetric higher alcohol process, they will be well advised in using Hewitt's salt as a de-aldehyding reagent. Our own conclusion regarding this matter is that there is only one process which is to any degree reliable, and that is the oxidation process. By modifying the conditions of working, the colorimetric process may be made to give almost any figure one pleases; and indeed, in practice, we have found that results returned by different observers (both using an iso-butylic control) have differed by more than 100 per cent.

DISCUSSION.

The PRESIDENT (Mr. Bevan) having invited discussion,

Dr. HEWITT said that he was pleased to hear that the authors intended to check the values obtained by the oxidation process in the case of butyl alcohol and propyl alcohol. To his mind this was a matter of absolute necessity, because, although by addition of brine to a specific gravity of 1·1 and extraction with carbon tetrachloride the whole of the amyl alcohol was certainly extracted, nevertheless there was no proof that the whole of the propyl alcohol that might be present would be extracted, since at ordinary temperatures propyl alcohol was miscible with water in all proportions, though probably at a lower temperature the mixture of propyl alcohol and water would separate out as amyl alcohol and water did at ordinary temperatures. With so great a difference in water solubility it seemed essential to know exactly what happened. He was very glad to find that the methyl orange titration for "mineral acid" could be dispensed with, in the first place because he found it rather difficult at any time to get the end-point with this indicator, which was not a very handy one to use. He did not think that he was altogether peculiar in this respect. In the second place, in dealing, as in this case, with an acid that was only very incompletely ionized, the end-point must of necessity be very far from sharp. He had never worked out the ratio, but it had always struck him that the more higher alcohols he found in a spirit, the more mineral acid apparently came over. This, of course, was what, on thinking over the matter, one would expect. With regard to the colorimetric method, he had himself used it in a great many cases, and he thought that probably others had too. Of course there were two ways of looking at it. From a scientific point of view it must be agreed that the colorimetric method was useless; but he did not feel quite sure that the possibility of its affording an index of quality had even now been entirely got rid of. The way in which errors arose was obvious from what the authors had said as to the cause of the coloration. The iso-butyl alcohol might be rectified, but with iso-amyl alcohol there was a difficulty, because in ordinary commercial iso-amyl alcohol there were the active alcohol boiling at just over 120° and the alcohol boiling at 131½°. Which of these two gave the stronger coloration was not known, and it would be very difficult for two workers to get specimens exactly alike. He had recently made some experiments in this direction, and had found it possible, by repeated fractionation of samples bought as pure, to get the higher fraction with the correct boiling-point within about ¼°. Probably that was fairly pure iso-butyl carbinol. It was, however, very easy to get small admixtures, the effect of which was unknown. No doubt the authors had worked in every case with the same iso-amyl alcohol, and had carefully purified it; but on looking at the results one found very varying ratios between the figures obtained with iso-butyl alcohol and with iso-amyl alcohol, whereas one would expect the ratio to be the same. He noticed that, using the reagent which he had proposed for the removal of aldehydes, the authors stated that higher results were, as a rule, obtained than with meta-phenylene-diamine hydrochloride. The result in the case of Experiment 4 in Table VII., however, was somewhat lower, and he thought that generally they were not very far apart. With regard to the experiments recorded in Table VIII., in which heating over a naked flame and on the water-bath for one hour were compared, the Allen-Marquardt figures in those cases were not given, but presumably there were very varying amounts of higher alcohols. With the naked flame the figures varied from 150 to 345, while on heating on the water-bath for one hour they all came out between 100 and 150. In order to be on the safe side he had generally, when he had used this method, heated for one hour on the water-bath, and he had obtained any number of figures between 100 and 150. The question of the removal of furfural was, of course, a very important one. In this connection the authors had mentioned the possibility of the formation of acridine dye-stuffs, which would be due probably to the formation of leuco-compounds, on which the strong sulphuric acid would act as an oxidizing agent. He had happened at one time to be interested in the acridines, and knew that they gave intensely coloured solutions.

Mr. CHAPMAN said that he, too, had been glad to hear that the authors proposed to extend their investigation to propyl alcohol. So far as his own experience had gone, whenever he had taken the precaution of determining the average molecular weight of the fatty acids by means of the barium salts, he had usually found this to indicate that they consisted almost entirely of butyric acid and valeric acid; in other words, there was, as a rule, very little of the oxidation product of propyl alcohol. He thought, therefore, that very small proportions of propyl alcohol were extracted in this method, and if that alcohol was to be included under the heading of "higher alcohols" it seemed quite possible that the results hitherto obtained were not by any means accurate. He thought that the great importance of the authors' two communications on this subject lay in the fact that they had gone far to establish the superiority of the Allen-Marquardt method over the others they had investigated. The surface-tension methods, the Beckmann method, the colorimetric method, and the Röse-Herzfeld method had all been, or were being, severely discredited, and whatever errors might be inherent in the Allen-Marquardt process, it was at least based on scientific principles, and gave results which in the hands of different observers were capable of being compared.

Dr. SCHIDROWITZ, in reply, said that he was inclined to agree with Mr. Chapman that the examination of propyl alcohol would indicate that some of the results obtained in the past were not quite accurate. He agreed with Dr. Hewitt that the end-point in the titration with methyl orange had been a source of difficulty. At the same time he thought it might be well to continue to do it roughly, to see whether the ratio they had mentioned existed. In cases in which the ratio was found to be abnormal, a gravimetric estimation of chlorine should be carried out. This, however, would in practice only very rarely be necessary. The utility of the colorimetric method even as an index of quality was, he thought, very fallacious. He had known whiskies of the same class to show enormous differences by the colorimetric method, while there was little difference in the results obtained by the Allen-Marquardt method, and the whiskies themselves were very similar in flavour, body, and all the other qualities upon which commercial value depended. The impurities to which such differences were due, and which undoubtedly were bodies other than higher alcohols, might be present in small or in relatively large quantities; but even if the quantities were relatively large, they were in many cases, in his opinion, insufficient to have any effect on the quality of the spirit. In the case in which Dr. Hewitt's reagent gave a low figure (namely, No. 4 in Table VII.) it was probable that there was only a very small quantity of these secondary constituents present, and that therefore the true value, as far as was possible by a colorimetric method, was obtained. At any rate, that reagent apparently took out the aldehydes and the furfural without doing anything else; but even in the case referred to, the meta-phenylene-diamine reagent probably was the cause of some secondary reaction. With regard to the purification of amylic alcohol and butylic alcohol, they had described in their last paper the purification of amylic alcohol, and the same applied to butylic alcohol. But when different specimens of commercial iso-butylic alcohol were most carefully purified by the method referred to, even though the resultant products displayed the same boiling-point, yet he knew from experience that their colorimetric properties were by no means necessarily identical; in fact, he had found wide differences.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Amount of Alcohol in Bread. Otto Pohl. (*Zeits. angew. Chem.*, 1906, xix., 668.)—The author has investigated the statement of Balas (*Dingl. Journ.*, ccix., 399) that fresh (English) bread contains from 0·2 to 0·4 per cent. of alcohol. He distilled large quantities (over 4 kilogms.) of two kinds of German bread with water, and found the bread made from wheat-flour, using stale dough, contained 0·0744 and 0·0830 gram of alcohol per 100 grams bread, and bread made from the same flour, using yeast, contained 0·0508 and 0·0547 gram of alcohol per 100 grams bread. Besides the alcohol, which was identified beyond all doubt, he always obtained a small quantity of a heavy dark-brown oil, possessing the characteristic odour of new bread. A. G. L.

The Volumetric Determination of Casein in Milk. H. V. Arny and T. M. Pratt. (*Amer. Journ. Pharm.*, 1906, vol. 78, pp. 121-128.)—A definite quantity (5 to 30 c.c.) of the milk is treated with 20 c.c. of $\frac{N}{10}$ ferric alum solution (48·1 grams per litre) at the ordinary temperature, the mixture diluted with water, allowed to stand for a few minutes and filtered, the filter washed, and the excess of ferric alum in the filtrate washings (about 200 c.c.) determined by titration with potassium iodide, acid, and thiosulphate, the difference between the amounts taken and found giving the quantity required for precipitation of the casein. Experimental results quoted show that the method yields concordant results, and that the fat and sugar in the milk do not affect the reaction. In almost every case 10 c.c. of milk required 4 c.c. of the reagent (0·1924 gram of ferric alum) for precipitation of the casein. Numerous comparative determinations were also made to establish the relationship between the amount of nitrogen in the precipitate and the amount of ferric alum solution consumed. The nitrogen determined by Kjeldahl's method in the precipitates from 10 c.c. of six different milks ranged from 0·0322 to 0·0372 gram, and this corresponded to 2·05 to 2·32 per cent. of casein (factor 6·25), or 2·1 to 2·37 per cent. with the factor 6·38. The results of duplicate determinations were in close agreement. A deposit may form in the clear filtrate from the casein after the liquid has stood for some time, but this is due to ferric albuminate and not to casein, and the results of the titration are the same, whether the liquid be titrated whilst cloudy or after a second filtration. C. A. M.

A Rapid Method of Determining Fat in Cocoa. Tschaplowitz. (*Zeit. anal. Chem.*, 1906, xlv., 231-235.)—Several grams of the finely-divided cocoa (sufficient to yield 1 or 2 grams of fat) are placed in a flask holding about 80 c.c., and having a long neck graduated from 73 to 77 c.c. in fifths of a c.c., and are boiled with 10 to 15 c.c. of alcohol, with constant shaking. The contents of the flask are then allowed to cool, 10 to 15 c.c. of ether introduced, and the mixture thoroughly shaken and boiled, after which it is cooled and made up to about 77 c.c. with ether, and the flask put aside. After about an hour or so the supernatant liquid will be sufficiently clear for 50 c.c. to be withdrawn with a pipette, a note being made of the total volume. This portion is evaporated in a porcelain basin on the water-bath, the residue taken up with a little warm ether and filtered through cotton-wool, the filtrate and washings evaporated, and the residue dried at 70° to 80° C., and finally for a short time at 100° C., and weighed. The deposit of fat-free cocoa in the flask has a density of nearly 1·6, and if 3 grams of cocoa were originally taken, 1 c.c. should be deducted from the total volume in the flask. The first boiling with alcohol appears to rupture the fat-cells completely. C. A. M.

Use of the Immersion Refractometer in the Determination of Caffeine. J. Hanus and K. Chocensky. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, pp. 313-320.)—The strength of aqueous solutions of caffeine may be determined by means of the immersion refractometer (*cf.* ANALYST, 1903, xxviii., 91), as the refraction is directly proportional to the quantity of caffeine present. At a temperature of 17·5° C. distilled water itself gives a scale reading of 15, and a solution containing 1 gram of caffeine in 100 c.c. of water a reading of 20. The amount of caffeine in 100 c.c. of solution may therefore be calculated from the formula,

$$x = \frac{y - 15}{5},$$

where y is the reading observed. A table is also given showing the amounts of caffeine corresponding with each 0·1 refractometer degree from 15 to 20.

W. P. S.

Determination of Sulphurous Acid in Meat. C. Mentzel. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 320-324.)—From the results given in this paper it is seen that meat itself, when acidified with phosphoric acid, distilled in an atmosphere of carbon dioxide, and the distillate oxidized with iodine and precipitated with barium chloride, never yields more than 0·002 per cent. of sulphur dioxide. The addition of onions to the meat (as is the case in some German potted meats) scarcely increases this amount, onions themselves only yielding 0·006 per cent. of sulphur dioxide when distilled under the above-mentioned conditions. The author concludes that meat or potted meat giving more than 0·004 per cent. of sulphur dioxide, or 0·005 per cent. in the case of samples flavoured with onions, must be considered as containing added sulphite preservative. W. P. S.

A New Method of Detecting Fluorine in Foods. J. Ville and E. Derrien. (*Bull. Soc. Chim.*, 1906, xxxv., 239-246.)—The method is based upon the fact that sodium fluoride modifies the spectrum of methæmaglobin, producing a new one characterized by an absorption band in the orange-red to the right of the methæmaglobin band. The reagent is prepared by mixing defibrinated ox-blood with four times its volume of a 0·1 per cent. solution of potassium oxalate, filtering the liquid, and dissolving a trace of potassium ferricyanide in 50 c.c. of the filtrate, in order to form methæmaglobin. In testing a liquid for fluorine, the reagent is added in the proportion of from 1 to 1·5 c.c. to 25 c.c. and the mixture examined with a spectroscope provided with a micrometer scale. In the case of wines the alcohol must be removed by evaporation, whilst red wines must be decolorized by means of precipitated manganese peroxide, which has also the effect of forming methæmaglobin in blood. Thus, on treating 25 c.c. of the filtrate with 0·1 gram of manganese peroxide, adding 1 to 1·5 c.c. of defibrinated blood mixed with four times its volume of 0·1 per cent. potassium oxalate solution, filtering, and examining the filtrate, the absorption spectrum is visible for about an hour when the wine contains from 0·08 to 0·1 gram of sodium fluoride per litre. It is rendered more pronounced and persistent by treating the wine successively with manganese peroxide and a solution of white of egg (to remove tannins) before adding the blood reagent. For the detection of fluorides in beer it is sufficient to test the liquid with the reagent after the removal of the alcohol and carbon dioxide, and the treatment with manganese peroxide and egg albumin is not required. Milk is examined by shaking 50 c.c. with about 4 c.c. of a 5 per cent. solution of oxalic acid, added drop by drop with constant shaking, heating the liquid for a few seconds on the water-bath, filtering it, and testing 25 c.c. of the filtrate with the reagent. As little as 0·05 to 0·1 gram of fluoride per litre of milk is readily detected, the characteristic absorption band remaining visible for several hours. In testing flesh foods a small portion of the sample is reduced to a pulp, and heated with a little water in a crucible on the water-bath. The process is repeated several times, the aqueous extracts cooled and filtered, and the filtrate mixed with the blood reagent and examined with the spectroscope. C. A. M.

A Dangerous Contamination of Phenacetin. C. Mannich. (*Ber. Deutsch. Pharm. Ges.*, xvi., 57; through *Pharm. Journ.*, 1906, lxxvi., 447.)—A sample of phenacetin that recently nearly caused the death of a patient was found to have a melting-point of 119° to 120° C. instead of 134° to 135° C. Further examination revealed the presence of 5·89 per cent. of chlorine in organic combination, and a substance was separated by digesting the sample with ether, evaporating the ethereal extract, and boiling out the residue with water. This substance proved to be *p*-chloracetanilide. Its occurrence in the sample could not be accounted for; it is not used in medicine, and is not formed during the preparation of phenacetin. The physiological action of *p*-chloracetanilide has not been determined, but the corresponding bromine compound, *p*-bromacetanilide, can be given internally with caution in doses of only 0·02 to 0·1 gram on account of the danger of producing collapse; *p*-chloracetanilide would probably act similarly. W. P. S.

The Alkaloids of Veratrum Album and their Determination. G. Bredemann. (*Apoth. Zeit.*, xxi., 41; through *Pharm Journ.*, 1906, vol. 76, p. 283.)—The rhizome and roots of *Veratrum album* (white hellebore) contain at least four alkaloids —viz., protoveratrine ($C_{32}H_{51}NO_{11}$), jervine ($C_{26}H_{37}NO_8$), pseudojervine ($C_{29}H_{43}NO_7$), and rubijervine ($C_{26}H_{43}NO_3$). Of these, the first is extremely toxic, jervine being weaker and the others non-toxic. The following method was employed for isolating the alkaloids: 5 kilogms. of the powdered drug were rendered alkaline with sodium hydroxide, exhausted with a mixture of equal volumes of ether and chloroform, and the ether-chloroform solution evaporated to a thin extract, which was extracted with dilute acetic acid. From the acid solution metaphosphoric acid precipitated the jervine and rubijervine. These were filtered off, and the filtrate made alkaline with ammonia and shaken out with ether, which extracted protoveratrine; it was then shaken out with chloroform, which removed pseudojervine. On treating the metaphosphate precipitate with sulphuric acid, rubijervine sulphate, being very soluble, was dissolved, leaving jervine sulphate as an insoluble residue. Commercial samples of the drug contained from 0·36 to 0·67 per cent. of total alkaloids. The alkaloids separated from three samples of the drug consisted of: Jervine, 0·402, 0·217, and 0·399; rubijervine, 0·053, 0·067, and 0·047; pseudojervine, 0·038, 0·032, and 0·027; protoveratrine, 0·062, 0·061, and 0·057 per cent. The mean neutralizing power of the alkaloids was found to be 0·00424 for each c.c. of $\frac{N}{10}$ acid, and the alkaloids may be determined volumetrically by adding 25 c.c. of $\frac{N}{10}$ acid to an aliquot part of the ether-chloroform solution, allowing about one-half the solvent to evaporate and titrating the excess of acid.

The following process may be used for the determination of the alkaloids in the tincture: 100 grams are evaporated to about one-half; 1 gram of solid paraffin and 25 c.c. of water are added and the evaporation continued until all the alcohol is removed. Two grams of acetic acid are now introduced, and the mixture stirred, cooled, and filtered. The separated cake is again warmed with 20 c.c. of water and 1 gram of acetic acid and filtered. The united filtrates are rendered alkaline with sodium hydroxide, shaken out with ether-chloroform mixture, and the solution obtained titrated as described above. Commercial tinctures examined yielded from 0·024 to 0·063 per cent. of alkaloid. W. P. S.

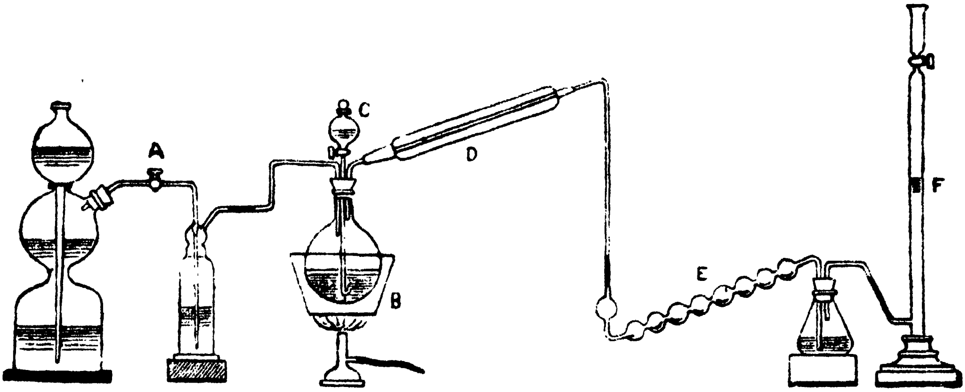
Analysis of Nitroglycerine Tablets. E. Sautesson. (*Svenska Farm. Tids.*; through *Pharm. Journ.*, 1906, lxxvi., 409.)—One hundred tablets are weighed, powdered, and extracted with ether in a Soxhlet apparatus. The residue obtained on evaporating the ethereal solution consists of cacao butter and nitroglycerine; it is saponified with alcoholic potassium hydroxide solution, and the potassium nitrate yielded by the nitroglycerine reduced by nascent hydrogen, the ammonia produced being then determined in the usual manner. W. P. S.

The Examination of Formaldehyde Pastilles. Ernst Rüst. (*Zeits. angew. Chem.*, 1906, xix., 474.)—The "formaldehyde pastilles" (trioxymethylene), used for disinfecting purposes, should weigh as nearly as possible 1 gram each, be practically completely soluble in hot water, and give a neutral solution. When tested by Blank and Finkenbeiner's method they should yield 95 to 97 per cent. of formaldehyde. Pastilles with only 85 per cent. of formaldehyde are found in commerce. They should be tested for carbonaceous residue by heating 10 grams in a platinum dish over a burner until they take fire, when they are allowed to burn themselves out without further heating; the residue should not exceed 0·1 per cent.; pastilles with a 0·5 per cent. residue are absolutely useless. The residue obtained should be examined for ash, which should not exceed 0·05 to 0·08 per cent., and give a neutral or only faintly alkaline reaction. The gases given off during the burning should also be observed, as the author has found pastilles which evolved what was apparently chlorine during the heating. A. G. L.

Effects of Borax and Boric Acid on the Human System: A Critical Review of the Report of Dr. H. W. Wiley. O. Liebreich (Pamphlet.)—The author reviews and criticises Dr. Wiley's report (*cf.* ANALYST, 1904, xxix., 357) on the effect of borax administered with food. From an exhaustive examination of the figures and reports, he concludes that no injurious effects were produced by the administration of the boron preservatives, and considers that the symptoms of ill-health noticed during the experiments must be attributed to inefficient hygienic conditions, to an injudicious mode of administering the preservative, and in a few cases to an unsuitable choice of persons for the experiment. The slight loss of weight of some of the men may be explained by chance occurrences at the preservative table; moreover, a loss of weight does not by any means always indicate an injurious influence. Liebreich also claims that the experiments on metabolism were undertaken with no equilibrium of nutrition, and that the fore periods were too short to prove regularity in feeding. As the percentages of nitrogen and phosphoric acid in the food were constantly changing, it is impossible to decide whether the excretion increased during the preservative period. He also considers that the medical supervision was insufficient, and that the situation of the dining-room left much to be desired. W. P. S.

ORGANIC ANALYSIS.

Quantitative Determination of the Carbonyl Group in Aldehydes, Ketones, etc. Watson Smith, Jun. (*Chem. News*, vol. 93, p. 83.)—The method is a modification of that of Strache, in which the aldehyde or ketone is mixed with an excess of phenylhydrazine, the excess oxidized by boiling with Fehling's solution, and the nitrogen gas liberated, collected, and measured. From 0·1 to 0·5 gram of the substance is mixed with 1 part, accurately measured, of a 5 per cent. phenylhydrazine solution, and 1·5 parts of a 10 per cent. sodium acetate solution, in a 100 c.c. measuring-flask. The bulk is made up with water to about 50 c.c., and heated on the water-bath for fifteen minutes, then cooled, diluted to 100 c.c., well shaken, and 50 c.c. pipetted into the dropping funnel, C, the stem of which is first filled with water. The end of the funnel is drawn out into a hook-shaped capillary, and dips down to the bottom of the flask, to prevent the collection of bubbles of gas. The flask has a capacity of 750 to 1,000 c.c., and contains 200 c.c. of Fehling's solution, on the surface of which floats a thin layer of paraffin wax. Between the flask and the nitrometer are a reflux condenser, to prevent any liquid distilling over, and an absorption apparatus, E, which removes the benzene vapour. Several substances have been tested as to their suitability as absorbing material for charging the apparatus, E, including paraffin oil and wax, caoutchouc, sulphuric acid, and a mixture of sulphuric and nitric acids in molecular proportions, the last being found to give the best results. The contents of the flask are first heated, while a current of well-washed carbon dioxide passes from the Kipp's apparatus through the flask and absorption bulbs to the nitrometer until the bubbles of gas rising in the latter are sufficiently small to be negligible. The solution in the funnel is then allowed to mix with the Fehling's solution, and the funnel rinsed out once or twice with hot water. The gas evolved by the reaction between the Fehling's solution and phenylhydrazine is carried along by the carbon dioxide through the absorption apparatus into the nitrometer, which is filled with potassium hydroxide solution (1 in 1). As soon as the bubbles have become as small as before the nitrometer is disconnected, allowed to stand some time, and the gas volume read off and reduced to 0° C. and 760 mm. Immediately prior to the actual experiment with the aldehyde or ketone a blank determination is made with the phenylhydrazine solution alone, 10 c.c. of which, with the requisite quantity of sodium acetate solution, is diluted to 100 c.c., and 50 c.c. pipetted into the dropping-funnel, and the process carried out as described above. The amount of nitrogen evolved in the actual determination deducted from the blank gives the volume of nitrogen equivalent to the phenylhydrazine used in forming a hydrazone with the aldehyde, 1 c.c. of nitrogen being equivalent to 0·001252 gram of CO. W. H. S.



Novel Reactions of Aceto-acetic Acid. E. Riegler. (*Münchener medizin. Wochenschr.*, 1906, vol. 53, p. 448; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 122.)—Arnold's diazo reaction is best carried out as follows: 20 c.c. of the urine are well mixed with 4 or 5 drops of concentrated hydrochloric acid and 10 c.c. of ether, 1 c.c. of a solution of 1 gram of *p*-amino-acetophenone and 5 c.c. of hydrochloric acid in 100 c.c. of water, 1 c.c. of a 0·5 per cent. sodium nitrite solution, and, after well shaking, 10 drops of 10 per cent. ammonia are added. After again shaking, the ethereal solution is separated; one part is evaporated to dryness, and the residue treated with 5 to 6 drops of hydrochloric acid; the remainder of the ethereal solution is shaken with concentrated hydrochloric acid. In both cases a fine bluish-violet colour is obtained if aceto-acetic acid was present in the urine. This colour is due to diazo-acetophenone diacetic acid—

$$CH_3.CO.C_6H_4.N:N.CH\left\langle\begin{matrix}CO.OH\\CO.CH_3\end{matrix}\right.,$$

a brown substance, insoluble in water, soluble in chloroform, alcohol, and ether. From its alcoholic solution this substance crystallizes in fine violet microscopical crystals. A. G. L.

Detection and Determination of Nitrotoluene in Nitrobenzene and of Toluene in Benzene. P. N. Raikow and E. Ürkewitsch.

(*Chem. Zeit.*, 1906. xxx., 295.)—The authors have found that potassium hydroxide forms a brown compound with both nitrotoluene and nitrobenzene, whilst sodium hydroxide reacts only with nitrotoluene. Rubidium hydroxide behaves similarly to potassium hydroxide, and lithium hydroxide resembles sodium hydroxide in its action, which is, however, less marked. Nitrotoluene can consequently be distinguished from nitrobenzene by treating it with sodium hydroxide. The test is best carried out by placing 0·5 gram of powdered sodium hydroxide and 1 c.c. of gasoline in a test-tube and allowing a small drop of the liquid to be tested to flow down the side of the tube. Even 0·00025 gram of nitrotoluene will produce a brownish colour, which may change to gray, reddish, or even blue in a few minutes, and is quite unaffected by any nitrobenzene present. By comparing the colour obtained with that given by a known amount of nitrotoluene the quantity of the latter may be determined with some degree of exactness.

Benzene may be examined for toluene by testing the nitrated oil as above. The authors find that it is practically impossible to remove toluene completely from benzene by fractional crystallization, but that pure benzene can be obtained by fractional nitration, toluene being more readily nitrated than benzene.

A. G. L.

The Determination of Suspended Water in Naphtha by Means of Wyleshinski's Centrifugal Machine. K. Charitschkow.

(*Westnikshiro w. weschtsch.*, 1905, vol. 6, p. 187; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 93.)—Very accurate determinations of the suspended water in naphtha can be made by mixing the sample with its own volume of benzene and then treating in Wyleshinski's centrifugal machine. The results are always a little higher than those obtained by diluting the sample with benzene and allowing the water to settle out of its own accord. A. G. L.

The Bromine Absorption of Oil of Turpentine. W. Vaubel.

(*Zeit. öffentl. Chem.*, 1906, xii., 107, 108.)—The following is an improvement on the author's original method of determining the bromine absorption of oils of turpentine: From 1 to 2 grams of the sample are dissolved in chloroform, about 100 c.c. of water, 5 grams of potassium bromide, and 10 c.c. of concentrated hydrochloric acid are added, and the mixture then titrated with a standardized potassium bromate solution until it shows the yellow colour of free bromine. The absorption of bromine is at first rapid, but becomes slower towards the end of the reaction; the bromate solution should be added until the coloration persists for half an hour. American oil of turpentine, according to the results given, has a bromine absorption of from 220 to 230, French 240, quantities approximating to that required for the formation of the tetrabromide, whilst pinene has an absorption of 254. As is shown in the author's table, turpentines are met with which nearly approach this last number; more generally they have absorption numbers of from 220 to 230; hence commercial turpentines contain from 86·6 to 90·5 per cent. of pinene, or some body having a similar bromine absorption. Many adulterants contain no pinene. Since some of the American turpentines polarize to the left, and the amount or kind of rotation gives no sufficient criterion as to their purity or otherwise, there only remains as reliable the bromine absorption test.

W. P. S.

Linaloe Oil. E. J. Parry and C. T. Bennett. (*Chemist and Druggist*, 1906, lxviii., 544.)—The authors have examined a number of samples of this oil obtained from the most trustworthy sources, and consider that the following figures fairly represent the analytical data yielded by the best quality of linaloe oil:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
Specific gravity at 15° C.	0·882	0·884	0·882	0·882	0·887	0·879
Optical rotation	- 13°	- 11°	- 11°	- 10°	- 11°30′	- 12°10′
Esters	11·2%	5·8%	—	5·5%	5·8%	5·5%
Linalol	57%	56·2%	—	63%	66%	65·5%

At least 55 per cent. of the oil should distil between 195° and 205° C., whilst usually over 60 per cent. is obtained. The optical rotation of the main fractions obtained from genuine oils scarcely differs from that of the oil itself, whilst the fractions yielded by adulterated samples possess widely differing rotations. The authors do not propose definite standards, but think that the above-mentioned figures may be of assistance in judging the purity of a sample of linaloe oil.

W. P. S.

Examination of Rosin Oil. Utz. (*Chem. Rev.*, 1906, xiii., 48; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 108.)—The author has examined a number of methods for the detection of mineral oils in rosin oils. He finds that the acetone method is useless for small quantities (5 to 10 per cent.) of mineral oil. Finkener's method (*Zeits. anal. Chem.*, 1887, 652) gave better results, 1 volume of rosin oil being soluble in at most 17 volumes of a mixture of 10 volumes of alcohol (specific gravity 0·8182 at 15·5° C.) and 1 volume of chloroform, whilst 1 volume of mineral oil does not dissolve even in 100 volumes of the mixture. Carbon tetrachloride cannot be used to separate the two kinds of oils, both being soluble in it. Holde's method (*Zeits. angew. Chem.*, 1891, 588) is useful. It consists in shaking the oil at the ordinary temperature with 96 per cent. alcohol, and determining the refractive index of the undissolved oil. Small quantities of mineral oil may be completely dissolved by the alcohol, but can be separated again by adding a little water. The presence of mineral oil lowers the refractive index so much that 1 per cent. of any mineral oil, excepting petroleum, can still be recognised in this way. The author believes it possible to work out a method which depends on treating the oil with concentrated and fuming sulphuric acid, and then determining the refractive index of the residual oil. A. G. L.

On the Use of Dimethyl Sulphate for the Detection and Determination of Coal-tar Oils in Mixtures with Rosin Oils and Mineral Oils, and on its Behaviour towards Fatty Oils, Spirits of Turpentine and Pinoline. E. Valenta. (*Chem. Zeit.*, 1906, xxx., 266.)—The author has found that dimethyl sulphate dissolves aromatic hydrocarbons, such as occur in coal-tar, in all proportions, whilst it neither dissolves, nor is soluble in, paraffin hydrocarbons or rosin oils. Consequently, the quantity of coal-tar oils added to mineral oils or rosin oils may be rapidly determined by simply shaking up the sample with one and one-half times its volume of dimethyl sulphate in a stoppered measuring cylinder, and noting the increase in volume of the dimethyl sulphate. The results obtained are accurate to 1 or at most 2 per cent. If desired, the oil can also be weighed by separating, destroying the dimethyl sulphate by saponification, and collecting the residual unsaponifiable oil as usual. Coal-tar oils may also be determined in a similar way in printing and lithographic inks, which contain linseed oil, rosin, etc., by first separating the pigment as usual, then saponifying with alcoholic caustic soda, extracting the unsaponifiable matter with ether, and treating it with dimethyl sulphate as above.

Dimethyl sulphate also dissolves substances such as nitrobenzene, nitro-naphthalene, etc., used as deblooming agents for mineral oils, and may be used for their extraction. On mixing dimethyl sulphate with fatty oils (olive, almond oils), its volume diminishes. The contrary is the case with pinoline and spirits of turpentine, about 30 per cent. of these bodies being dissolved in the dimethyl sulphate. Pinoline yields an orange solution in the cold; on heating, it dissolves completely, forming a dark-gray liquid; the mixing is unaccompanied by any rise in temperature. Turpentine gives a colourless solution and a marked rise in temperature. Dimethyl sulphate possesses toxic properties, and should not be handled too freely. A. G. L.

The Examination of Raw Caoutchouc. W. Esch. (*Gummi Zeit.*, 1906, vol. 20, p. 494; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 101.)—The raw caoutchouc is separated by testing with the finger into firm elastic and soft or tarry. Dirt and impurities should be looked for both with the naked eye and a lens, and it should be remembered that a quantity of dirt which would absolutely condemn the use of a soft tarry kind for many purposes can be easily washed out of a firm elastic variety. Cuts should be made in the rubber, and the colour noted; it should be lighter than that of the exterior. If moisture or serum ooze out, they should be tested to see whether they will yield caoutchouc. Failure to do this indicates a faulty coagulation of the sample.

The author insists that the actual chemical examination *must* be made on the washed and dried samples only, not on the raw caoutchouc, and gives some further details concerning his and Chwolles' method. A. G. L.

The Volumetric Determination of Uric Acid. A. Ronchèse. (*Journ. Pharm. Chim.*, 1906, xxiii., 336-340.)—Uric acid is oxidized by iodine at the ordinary temperature in a medium rendered alkaline by potassium bicarbonate, borax, or other substance without action upon iodine, each molecule of uric acid requiring two atoms of iodine. Thus each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0·0084 gram of uric acid. In using the method with urines, it is necessary to separate the uric acid from other bodies that might act upon the iodine—*e.g.*, by precipitating it as ammonium urate in the following manner: 100 c.c. of the urine are treated with 15 c.c. of ammonium hydroxide and 15 grams of ammonium chloride, and the mixture allowed to stand for thirty minutes. The precipitated urate is collected and washed with a solution containing 150 c.c. of ammonium hydroxide and 150 grams of ammonium chloride per litre, and then suspended in 300 c.c. of water and dissolved by the addition of dilute acetic acid. The solution is now rendered strongly alkaline by the addition of 20 c.c. of a saturated solution of potassium bicarbonate or borax, and titrated with a $\frac{N}{10}$ solution of iodine. The number of c.c. used, multiplied by 0·084, plus 0·01 gram (the correction for the solubility of the precipitate during the washing), gives the amount of uric acid per litre. The results thus obtained are in close agreement with those given by the Salkowski-Ludwig method. The presence of albumin has no appreciable effect upon the determination. C. A. M.

The Determination of Small Quantities of Ammonia in the Presence of Urea. Frenkel. (*Bull. Soc. Chim.*, 1906, xxxv., 250, 251.)—The drawback of Schlösing's method of liberating the ammonia by means of calcium hydroxide in the cold is that as long as sixty hours are required for complete absorption of the ammonia in the standard acid. The author, however, finds that a determination can be made in two hours by passing a current of air through the flask, the air itself having first been freed from ammonia by passage through sulphuric acid, and from carbon dioxide by passage through potassium hydroxide solution. Test results quoted show that the whole of the ammonia is expelled from the solution under examination within two hours, and that no fresh ammonia is produced from the urea.

C. A. M.

Rosin Size Analysis. Clayton Beadle and Henry P. Stevens. (*Chem. News*, 1906, vol. 93, p. 155.)—In the usual method of determining moisture in rosin size, which consists in determining the loss in weight when the size is heated in a flat dish or clock-glass at 100° to 110° C., any spirit of turpentine left in the rosin may volatilize and cause too high a result. To see whether this error is serious, the authors have determined the moisture in other ways: (1) The size was heated in a glass flask placed in an air-bath; the flask was connected to a condensing tube, the end of which was bent down and dipped for a short distance into a burette previously filled up to the 50 c.c. mark with water; the volume of water distilled over, as well as that of the turpentine, was directly read off. (2) The loss in weight of the flask used in method (1) was noted. (3) Water was determined by difference after determining total rosin, total soda, turpentine, and other mineral matter in the size. The mean values obtained by the different methods on a sample of size containing 0·34 per cent. of turpentine were: (1) 38·75, (2) 39·55, (3) 39·84 per cent. of moisture. The usual method gave 39·5 per cent. The authors infer that for samples of size containing only small amounts of turpentine the usual method is sufficiently accurate.

A. G. L.

On Methods of Distinguishing between the Pitch from Brown Coal-tar and other Pitches. E. Graefe. (*Chem. Zeit.*, 1906, xxx., 298.)—Pitch from brown coal-tar, including both the "goudron" left by the distillation of the tar and the acid-pitch obtained in the purification of the distillates with sulphuric acid and soda, can be distinguished from most other kinds of pitch by its high content of phenols. This may be easily recognised by boiling 0·2 to 0·5 gram of the pitch with 5 to 10 c.c. of normal sodium hydrate solution, filtering, and adding to this solution a few drops of a solution of diazo-benzene chloride. The appearance of a red colour or precipitate indicates the presence of phenols. Pitch from wood-tar also contains an appreciable quantity of phenols, but may be recognised by the acidity of the vapours given off on distillation and by the incomplete solubility in benzene.

For the determination of the melting-point of pitches fusing at a high temperature (200° C.), the author modifies Krämer and Sarnow's method as follows: One end of a melting-point tube is closed by a cork; the usual quantity of pitch is then placed in the tube and covered with a layer of 5 grams of mercury. The pitch is melted over a naked flame or in a paraffin bath, and allowed to cool. The cork is then removed, and the tube heated as usual in a bath of paraffin, the layer of air below the pitch protecting the latter from the solvent action of the paraffin on it.

A. G. L.

Examination of Paints. J. R. Moechel. (*Chem. Trade Journ.*, 1906, xxxviii., 235.)—Of twenty tests, the first eight, which were applied to plates of glass, wood, tin-sheeting, galvanized iron, or sheet steel, 1 inch by 3 inches or 2 inches by 5 inches, coated with the paint and allowed to dry for eight days at the ordinary temperature, determine the resistance to various reagents—viz. (1) *acids*, by immersion in 10 and 25 per cent. HCl, H_2SO_4, and HNO_3, for twenty-four hours at room temperature, and a second series for two hours on the water-bath; (2) *acid fumes*, by allowing the fumes of hot HCl, H_2SO_4, and HNO_3 to play directly on the paint for three minutes; (3) *alkalies*, by immersion in 10 and 25 per cent. NaOH, both at room temperature, and on the water-bath for fifteen minutes, noting appearance after standing one hour; (4) *ammonia fumes*, by blowing air for five minutes into a wash-bottle one-fourth full of strong NH_4OH, having a short stem funnel through the cork, on which is laid the painted surface; (5 and 6) *sulphurous fumes* and *hydrogen sulphide*, by treating the painted surface for thirty minutes with SO_2 or H_2S, as described with NH_4OH; (7) *steam under pressure*, by exposure for one hour at 18 to 20 inches from the boiler safety-valve; and (8) *free steam*, by subjecting for twelve hours to steam from a water-bath. The *spreading* and *covering capacities* are measured respectively by the amount of paint used, and the number of coats required to obtain opaqueness. The *rust-preventing power* is determined by applying a double coat to plates of sheet-iron, thoroughly polished with a dry powder, allowing to dry for eight days, and then loosening the coat of paint by placing the plates face downwards upon a boiling water-bath for about twelve hours, drying with filter-paper, and finally for one hour at 100° C., employing a little $CHCl_3$, if necessary, to help loosen the edges. With good paints no rust should be found on the polished metal. *Time of drying* should be observed, and the *elasticity* tested by bending and rebending painted strips of tin sheeting and thin, flexible drawing-paper, covering them with a glass plate, and weighing down for twenty-four hours with a block weighing 3 to 5 pounds. The *penetration into wood* is ascertained by applying two coats of paint to strips of white pine-wood, some air-dried, others dried in the water-oven for three hours, cutting the strips crosswise and lengthwise, and examining the sections with a good lens, and the power of *preventing absorption of water* by painting air-dry blocks of the material to be rendered waterproof, weighing them, and then boiling in distilled water for thirty minutes, drying with blotting-paper, and again weighing. *Adhesion* of the paint is tested by applying a vacuum to the surface, and the *fineness of pigment* by placing a small amount upon a glass plate, pressing down with another glass plate, standing both plates on end and allowing the paint to run, when, after one hour, no separation of pigment particles should appear for at least an inch of downward surface. The exact *shade and tint of colour* are examined by comparison with standards on microscopic glass slides, the two colours being placed side by side just touching each other, and viewed first over a white, then over a black surface. Finally, the effects of *dry heat* and of a *naked flame* are observed, the former by subjecting painted glass, wood, or metal plates to temperatures of 100°, 300°, 500° C., for one hour each, and the latter by exposing the painted surface to various gas-burners, of approximately known temperatures, for periods of one, two, and three minutes. W. H. S.

INORGANIC ANALYSIS.

On the Iodometric Determination of Copper. Paul Gerlinger. (*Zeits. angew. Chem.*, 1906, xix., 520.)—In order to obviate the uncertainty of the end-point in the iodometric determination of copper due to the presence in the liquid of a large precipitate, the author adds a sufficiently great excess of potassium iodide to redissolve the whole of the cuprous iodide first precipitated. For 0·2 gram of copper, at least 10 grams of potassium iodide (added in the powdered state) are necessary, and the bulk of the liquid should be kept low. He also finds it unnecessary to boil the liquid after the neutralization of the acid liquid by ammonia, as recommended by Low, provided no lower oxides of nitrogen are present. Working on pure copper solutions, the author obtained very exact values by his method. A. G. L.

On the Determination of Copper as Thiocyanate. Ernst Murmann. (*Oesterr. Chem. Zeit.*, 1906, ix., 67.)—The author has obtained good results by weighing the cuprous thiocyanate dried at 100° C., in a Gooch crucible. Large excesses of thiocyanate and of sulphurous acid should be avoided, and the precipitate should be washed first with a liquid containing potassium thiocyanate and sulphurous acid, and finally with a little alcohol. A. G. L.

Detection of Traces of Copper in Distilled Water. K. Ebert. (*Apoth. Zeit.*, 1905, xx., 908; through *Pharm. Journ.*, 1906, lxxvi., 387.)—About 500 c.c. of the water are treated with a few drops of ammonia, and then filtered three times through a plug of cotton-wool. A faint green colour on the cotton-wool indicates the presence of copper in quantity too small to be detected by hydrogen sulphide or potassium ferrocyanide. W. P. S.

Comparison of Wet and Dry Assay of Gold Telluric Ores. W. F. Hillebrand and E. T. Allen. (*Chem. News*, 1906, vol. 93, pp. 100, 101, 109-111, 121, 122, and 132-134.)—Working with ores containing 0·07 to 0·09 per cent. tellurium and 0·05 to 0·06 per cent. gold, the authors find that, provided slag and cupel losses are corrected for, fire assay by crucible gives results equal to those obtained by precipitating the gold by ferrous sulphate, after extracting with nitric acid, filtering, adding bromine water, evaporating nearly to dryness, digesting the residue with HCl, and evaporating two or three times with further quantities of HCl. An investigation of the errors occurring in fire assaying and parting, made on proof gold and silver from the United States Mint, has shown that the slag loss is very small, as is also the cupellation loss by volatilization except at high temperatures. The loss of gold by absorption is considerable, being largely influenced by slight changes in temperature, and greater with pure gold and alloys poor in silver than with those containing much silver. A higher temperature at the end of cupellation with gold beads than with silver is unnecessary, the most exact results being obtained when feather litharge was still abundant at time of brightening, and nothing is gained by leaving gold beads in the muffle for some time after brightening in order to remove last traces of lead. The error due to retention of lead in the beads is important, 0·30 and 0·37 per cent. of lead being found respectively in two cases. Silver is completely removed from quartation alloys by nitric acid, but only after three or more extractions. Admixture of pure nitrous acid with the nitric acid used in parting has no appreciable effect, and any slight trace of gold dissolved by pure nitric acid is negligible. W. H. S.

On the Separation of Tungsten from Tin. Ed. Donath. (*Zeits. angew. Chem.*, 1906, xix., 473.)—The recent publication of Angenot's process (ANALYST, this vol., p. 84) has induced the author to call attention to his own previously described and simpler method of effecting this separation. The mixture of stannic oxide and tungsten trioxide is heated to redness for fifteen minutes with zinc dust or filings in a covered porcelain crucible. The porous mass obtained is transferred to a beaker and warmed with dilute hydrochloric acid (1 to 2) until all the tin has dissolved, when the liquid is cooled somewhat and cautiously treated with powdered potassium chlorate until the blue tungsten oxide has been completely oxidized to yellow tungstic acid. After diluting the liquid with at least one and one-half times its volume of water, it is allowed to stand for twenty-four hours, when the tungstic acid is filtered off, washed first with water containing nitric acid, then with water containing ammonium nitrate, ignited and weighed. If necessary, tin can be determined, as usual, in the filtrate.

A. G. L.

The Direct Titration of Zinc by Means of Potassium Ferrocyanide. E. Mürmann. (*Zeit. anal. Chem.*, 1906, xlv., 174-181.)—The drawback of the ordinary method in which the end-point of the reaction is determined by "spotting" can be obviated by adding 2 to 5 c.c. of a 1 per cent. solution of uranyl chloride to the hot solution (100 to 150 c.c.). The change of colour from brown to bluish-gray is very sharp when the solution is nearly boiling, and 0·1 c.c. of the ordinary zinc solution (0·01 gram per c.c.) is sufficient to produce it when the excess of potassium ferrocyanide is titrated back. The ferrocyanide solution is prepared by dissolving 43·11 grams of the pure crystalline salt in a litre. Experiments to determine the influence of salts and acids showed that considerable quantities of salts, including ammonium chloride, cause only a slight increase in the amount of potassium ferrocyanide consumed, and that the effect of hydrochloric acid is also small, provided its quantity does not exceed 20 c.c. (of specific gravity 1·075) to 100 to 150 c.c. of liquid. Free nitric acid must not be present, and it is therefore advisable to use uranyl chloride instead of uranyl nitrate. For the determination of zinc in zinc blend, 2·5 grams of the sample are dissolved in hydrochloric acid with the addition of a little nitric acid, the solution filtered, evaporated with sulphuric acid, filtered, treated with hydrogen sulphide, filtered, and oxidized with bromine. It is then filtered into a 500 c.c. flask containing 100 c.c. of ammonia solution and 10 c.c. of concentrated ammonium carbonate solution, again filtered, and finally made up to the mark. A definite quantity is taken, treated with 5 c.c. of the uranyl chloride solution, and, after neutralization, with 10 c.c. of hydrochloric acid (specific gravity 1·075) titrated at an almost boiling temperature.

C. A. M.

The Determination of Manganese in Cast Iron and Steel. O. Brichant. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 124-127.)—The following modification of Schmidt's ammonium persulphate method is recommended as the most suitable for laboratory work. It is based upon the conversion of the manganese into permanganic acid by ammonium persulphate in the presence of silver nitrate:

$$(NH_4)_2S_2O_8 + 2AgNO_3 = Ag_2S_2O_8 + 2NH_4NO_3,$$
$$Ag_2S_2O_8 + 2H_2O = Ag_2O_2 + 2H_2SO_4,$$
$$3Ag_2O_2 + Mn(NO_3)_2 + 4HNO_3 = HMnO_4 + 6AgNO_3 + 2H_2O.$$

(*a*) *Steels.*—One gram of steel filings is dissolved in nitric acid of specific gravity 1·135, and the solution diluted with sufficient cold water to bring the temperature to about 50° C., and then treated with 10 c.c. of a $\frac{N}{10}$ solution of silver nitrate and about 0·2 gram of ammonium persulphate, and heated until the rose colour disappears. The liquid is then decolorized with ferrous sulphate solution, cooled to about 60° C., treated with a fresh portion of ammonium persulphate, shaken, and allowed to stand for about ten minutes. The whole of the manganese will then have been converted into permanganic acid, and the liquid, when cold, is titrated with standard sodium arsenite solution until the violet colour disappears.

(*b*) *Cast Iron containing less than 2 per Cent. of Manganese.*—One gram is dissolved in 25 c.c. of nitric acid, the solution diluted with cold water and filtered from silica and graphite, and the manganese determined as in the case of steel.

(*c*) *Spiegel and Ferro-Manganese.*—One gram is dissolved, the solution made up to 250 or 500 c.c., and the manganese determined in 25 or 50 c.c., after the addition of 25 c.c. of nitric acid.

The sodium arsenite solution is prepared by dissolving 0·9 gram of arsenious acid and 3 grams of sodium bicarbonate in 250 c.c. of water and diluting the solution to a litre. It is standardized by dissolving 0·278 gram of manganese dioxide in 15 c.c. of strong hydrochloric acid, adding 2 to 3 c.c. of strong sulphuric acid and evaporating the solution until the appearance of white fumes, after which it is cooled and made up to a litre with water. Fifty c.c. of this solution (=0·01 gram of manganese) are treated with 10 c.c. of silver nitrate solution (17 grams per litre), 25 c.c. of nitric acid (specific gravity 1·20), and about 1 gram of ammonium persulphate, and the whole heated to about 60° C., cooled, and titrated. The method is stated to be more rapid than the expensive bismuth tetroxide method, and to yield very accurate results.

C. A. M.

Derangement of a Public Water-Supply by Manganese Sulphate. R. Woy. (*Zeit. öffentl. Chem.*, 1906, xii., 121-125.)—Up to the beginning of the year 1905 the town of Breslau, Silesia, had been supplied with water drawn from the river Oder and filtered through gravel. From that time, however, a new and apparently more satisfactory supply was obtained from the surrounding country, the use of the filtered river-water being abandoned. About March 28 of this year an extraordinary change in the character of the water was noticed, the peculiarity first noticed being an unpleasant taste. On analysis the water was found to have a slightly acid reaction, and to contain a considerable quantity of manganese, the normal amount of sulphates in the water also showing an increase. The quantity of manganese present varied from day, the maximum found being 0·109 gram of manganous oxide (MnO) per litre. The source of the manganese salts has not yet been investigated.

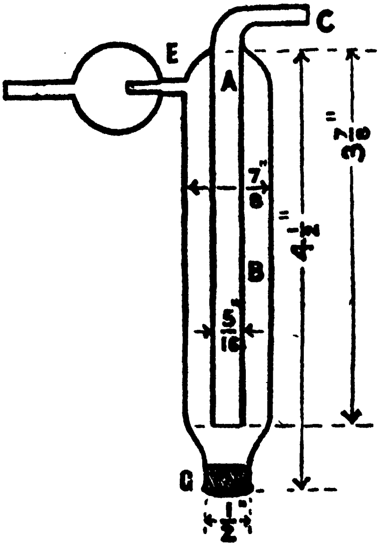
W. P. S.

On the Quantitative Determination of Cyanate in the Presence of Cyanide. Wilhelm Wild. (*Zeits. anal. Chem.*, 1906, vol. xlix., p. 122.)—Confirming Ewan's statement (*Journ. Soc. Chem. Ind.*, xxiii., 244), the author finds that the solubility of silver cyanate in pure water is very considerable, amounting to 0·0075 gram in 100 c.c. of water at 22° C. In solutions containing excess of silver nitrate, however, the solubility is very much less, being only 0·0006 gram in 100 c.c. of approximately $\frac{N}{100}$ silver nitrate solution. He consequently proposes to determine cyanate in the presence of cyanide by precipitating with an excess of standard silver nitrate in neutral solution, making the liquid up to a definite volume, and titrating the excess of silver present in an aliquot part of the filtered solution. On another portion of the sample the cyanide only is determined by precipitation with silver nitrate in nitric acid solution, the difference between the two values being taken as due to cyanate. Any carbonate present in the sample is removed by calcium nitrate before making the first titration. A. G. L.

The Preparation of Pure Chloric Acid for Analytical Purposes. V. Bernard. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 81, 82.)—A solution of 800 grams of pure barium chlorate in 1,700 c.c. of water is treated with 243 grams of pure sulphuric acid (60° Bé.) added little by little, whilst the flask is cooled in a current of water to prevent the liquid becoming hot, with the consequent formation of perchloric acid. The barium sulphate is removed by filtering the liquid through purified asbestos with the aid of a pump, and the almost colourless filtrate (1,750 c.c.) can be used for oxidizing purposes in the place of nitric acid or potassium chlorate. It has the advantage of being decomposed without residue in the presence of hydrochloric acid. C. A. M.

APPARATUS.

New Form of Calcium Chloride Tube. A. E. Hill. (*Proc. Chem. Soc.*, 1906, vol. 22, p. 87.)—The apparatus consists essentially of an inner exit-tube, A, fused into a larger tube, B, which has a side-tube, E, provided with the usual form of bulb. Both tubes are filled from the bottom, G, with finely-granulated calcium chloride, above and below which are placed plugs of glass wool, and the whole closed with a cork, which is cut off flush with the bottom of the tube and covered with sealing-wax. The space in B, above the side-tube, E, is left empty, to allow diffusion of the water vapour before it comes in contact with the calcium chloride, thus insuring uniform wetting of the latter. The advantages of the tube are (1) a double scrubbing action obtained with a comparatively small amount of calcium chloride; (2) its compactness and strength, there being no fragile bend, as in a U-tube; and (3) the ease of emptying and refilling, and of drying before weighing. The apparatus is manufactured by Baird and Tatlock, who have registered the design. W. H. S.



Magnesia Apparatus at High Temperatures. E. Wedekind. (*Chem. Zeit.*, 1906, xxx., 329.)—The author gives the results of some experiments on the behaviour at high temperatures of articles made of magnesia supplied by the Royal Berlin Porcelain Factory. He found that they stood the action of the electric arc well, fusing only at the parts actually touched by the arc, but would not stand in fused thermite. They also require careful heating and cooling if they are not to crack. The author believes that magnesia articles will not come into general use owing to their high price, coupled with the fact that as a rule they can only be used once, as fused substances adhere to them. For experiments with the arc he strongly recommends blocks of magnesite bound with iron, as supplied by the Frankfurter Gold und Silberscheideanstalt. These may be used up to sixty times before being destroyed.

A. G. L.

Delivery Funnel for introducing Liquids under Increased or Diminished Pressure. T. J. Bryan. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 80.)—Three forms of funnel are described, the principle being the same in each—viz., that on opening the tap the pressure on the upper and lower surfaces of the liquid in the funnel shall be the same, whether greater than that of the atmosphere, due to increase of pressure in the generating flask, or less, as in fractional distillation *in vacuo*.

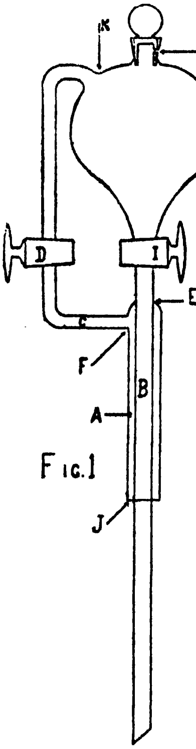


FIG. 1

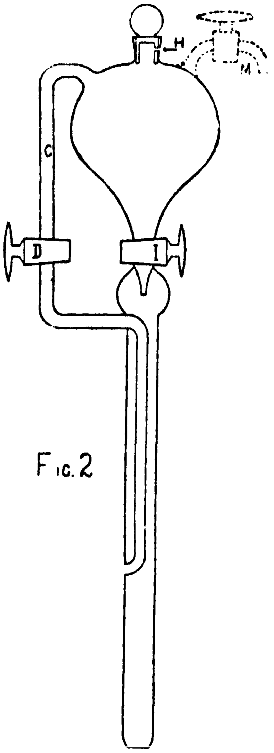


FIG. 2

Fig. 1 shows a dropping funnel, whose tube, B, which should be of small bore, and not exceed 4 inches in length, passes through and is sealed to the tube, A, at E, the side-tube, C, with a glass tap at D, being fused into the funnel at K, and into the tube, A, at F. The funnel has an air-vent in the stopper at H, and is fitted into the flask by means of a stopper passed over the tube, A. When I and H are closed, and D is open, the pressure existing in the flask is exerted through A and C upon the upper surface of the liquid in the funnel; and on opening I, the same pressure being applied to its lower surface also, the liquid flows down through B, under a pressure dependent only on its specific gravity, the height of its column, and the capillarity of the tube, B, which last may be neglected. The speed of delivery may be regulated by the tap, and a continuous or intermittent flow of liquid is possible so long as a single drop remains in the funnel. By closing D and I, and removing the stopper, fresh liquid may be introduced into the funnel.

Fig. 2 is a modification of Fig. 1, fitted with a Walter's dropping device, and the side, M, is useful when it is required to replace air in the apparatus with some other gas, or when it is undesirable to discontinue distillation *in vacuo* after refilling the funnel, in which case the funnel is exhausted through M, while D, I, and H are closed.

A cheaper form of the apparatus may be obtained, suitable for cases in which the reagents employed do not attack rubber, this latter being employed for making joints, etc. W. H .S.

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REVIEW.

Food Inspection and Analysis for the Use of Public Analysts, Health Officers, Sanitary Chemists, and Food Economists. By Albert E. Leach, S.B. (New York: Wiley and Sons. London: Chapman and Hall. Price 31s. 6d.)

The scope of this work is wider than that of many of the books on food analysis published in this country, embracing other matters relating to the control of the food-supply in the United States of America. The book seems well adapted to meet the wants of those for whom chiefly it has been written. It is copiously supplied with references, especially as regards American publications, and also German; but as regards British work and publications, the references are certainly less complete.

In the author's country the working of the Food Acts is in some respects in advance of what, so far. has obtained here. Many of the States have in local laws adopted standards for milk and other articles, while Congress in 1903 empowered the Secretary for Agriculture to draw up from time to time schedules giving limits and standards for many articles of food. American writers are apt to assume that we are in like position, and hence the author speaks of British standards for vinegar and other articles, which at present do not exist.

In the preliminary chapters the duties of the Food Inspector and of the Public or Official Analyst are considered, also the fitting and equipment of the food laboratory, followed by a general classification of foods. The system of inspector's lockers, described on p. 7, may be suggestive to our home authorities.

Under the fitting of the laboratory a formula is given for the aniline black staining of laboratory tables, where the aniline solution is too strong for use. Sixty-five grams of water probably means 615 grams, as in the second solution (pp. 13 and 14).

The description of general analytical methods is fairly complete, but those for the estimation of arsenic might have been written before the arsenic scare here in 1900. The great advances then made in the application of the Marsh test are ignored, and the mirror test method is ascribed to Sanger in 1890, though he used for comparison test solutions of arsenic, 0·01 mgm. As in 1 c.c. (or 1 of As in 100,000 of solution). In the modern methods, 1, 2, 3, etc., c.c. of such a solution would give mirrors so dense as to be indistinguishable, unless only a small fraction of the arsenic was deposited in the mirror. Tests capable of detecting $\frac{1}{500}$ or even $\frac{1}{1000}$ grain of As per gallon must be some hundreds of times more delicate than the method described by the author.

In subsequent chapters no mention is made of the possible presence of arsenic in malt derived from the fuel used in the malt-kilns. The presence of arsenic in vinegar is referred to, but not in other organic acids, nor in glycerine.

A very full account is given of milk and milk products. Obviously the minimum water in milk, given as 89·32 per cent., is a misprint for a lower number. One page is devoted to the "so-called" Adams' method, quoted without reference, while five pages are allotted to the Wollny Milk Fat Refractometer. This surely cannot indicate their relative importance.

In the estimation of milk-sugar, the Laurent instrument is referred to almost equally with the Soleil-Ventzke, but in the chapter on sugar the latter only is described in its various forms. In this chapter an error in the conversion table is noticeable which I find in other American books. 1° angular rotation is not equal to 0·7511° Wild's (sugar scale), but to ten times as much—7·511°.

Butter is relegated mainly to the chapter on edible oils and fats. While Hehner and Angell's names are given in the list of references at the end of the chapter, only ten lines are given at the beginning of the chapter to their pioneer work. The author speaks of the Reichert-Meissl number as "conclusive." Unfortunately, extended experience does not show any one test as being quite conclusive.

Phytosterol and cholesterol are referred to and described, but no mention is made of the acetate method for the identification of phytosterol, nor is any reference made to the possible effect of the feeding of the animals on the milk and other products obtained from them.

The chapters on flesh foods and eggs give much information useful to meat and market inspectors. The detection of preservatives in meats, fish, etc., is indicated, also the distinctive characters of horseflesh, but nothing is said about the vexed question of the influence of tuberculosis or other germ diseases on the wholesomeness of the milk and products from animals so infected.

There are interesting chapters on cereals, vegetables, and fruits; on tea, coffee, and cocoa, and on spices. These are freely illustrated both in the text and in numerous plates at the end of the book. Some of the illustrations are wanting in clearness, but they indicate what each analyst should do for himself—prepare his own slides of pure materials and mixtures for comparison, without relying too much on any book illustrations.

Chapters on alcoholic beverages, vinegar, food preservatives, and tinned and canned foods, are also included. On p. 530 the specific gravity of a de-alcoholized liquor is given as 0·9872. If this is compared with water = 1, there is obviously some mistake. Probably the chapter on alcoholic liquids was written before so much attention was drawn to the subject by prosecutions for selling impure spirits, and this may explain why only the Rose method for the detection and estimation of the higher alcohols is given in the text.

On the whole, the book may be recommended as giving a good account of the experience and practice of American laboratories in the analysis and examination of food.

T. F.

JULY, 1906. Vol. XXXI., No. 364.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Thursday evening, June 14, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. W. Dickson and G. W. Monier-Williams, B.A., Ph.D., were read for the second time.

Messrs. H. G. Harrison, M.A., E. Miller, and F. W. Passmore, Ph.D., were elected members of the Society.

The following papers were read: "An Examination of the Methods of Milk Analysis used at the Government Laboratory in Connection with Samples of Milk referred under the Sale of Food and Drugs Acts," by H. Droop Richmond and E. H. Miller; "On the Examination of Linseed, Olive, and other Oils," by R. T. Thomson and H. Dunlop; and "On the Composition and Valuation of Oils for Gas-making Purposes," by Raymond Ross and J. P. Leather.

* * * * *

A NEW METHOD OF ESTIMATING MOISTURE, WITH SPECIAL APPLICATION TO MOISTURE IN CORDITE AND OTHER SUBSTANCES CONTAINING VOLATILE MATTERS OTHER THAN WATER.

BY P. V. DUPRÉ.

(*Read at the Meeting, April* 4, 1906.)

IN a paper on ammonium oxalate, which I had the honour to read before this Society in June last, a method of estimating moisture by means of calcium carbide was mentioned, and it was stated that experiments were in progress to apply the method more or less generally. These experiments having been now completed, I beg to lay the results before this Society.

A series of experiments was first undertaken to standardize the process. With this object ammonium oxalate was chosen, as affording the most convenient and accurate method of weighing out a definite quantity of water, since the proportion it contains is accurately known (12·67 per cent.), and there is no tendency to loss by evaporation during weighing.

Various weights of the salt, from 0·25 to 0·56 gram, equivalent to 0·03 to 0·07 gram of water, were taken, the quantity being weighed out in a tube of about 1 cm. diameter and about 12 cms. long; the salt was then covered with a thin layer of sand,* and calcium carbide filled in to a depth of about 5 cms. The tube was then connected with a nitrometer containing a saturated solution of common salt, and the apparatus adjusted to zero with the tube brought to a known temperature by immersion in water. The tube was then immersed in a water-bath at a temperature of 100° C. to a depth of 7 or 8 cms., and the heating continued until no further evolution of gas could be observed. The tube was then brought back to the original temperature and the volume of acetylene measured, the temperature and pressure being noted.

As the mean of ten experiments, in the last three of which the water was weighed out direct, it was found that 1 c.c. of acetylene at N.T.P. was equivalent to 0·001725 gram of water. The theoretical number would be 0·00162, but at the temperature of boiling water the hydrate of lime formed retains a small proportion of water above the one molecule. As, however, the amount of hydrate of lime formed is always proportional to the amount of acetylene evolved, the relation between the amount of acetylene produced and the excess of water retained is always the same.

The following table gives the results obtained; it will be seen that the variations are small, and that the process is therefore of adequate accuracy:

Weight of Amount of Oxygen taken.	Weight of Water.	Amount found.	Error.
0·2430 gram	0·0308	0·0311	+0·0003
0·3003 ,,	0·0380	0·0378	-0·0002
0·3160 ,,	0·0401	0·0400	-0·0001
0·3235 ,,	0·0410	0·0413	+0·0003
0·4890 ,,	0·0620	0·0619	-0·0002
0·5020 ,,	0·0636	0·0634	-0·0002
0·5565 ,,	0·0705	0·0703	-0·0002
	0·0651	0·0649	-0·0002
	0·0298	0·0301	+0·0003
	0·0627	0·0625	+0·0002

The moisture in cordite was then estimated in exactly the same way, cordite, ground as for analysis, being substituted for the oxalate, and from 1 to 2·5 grams being taken. The total volatile matter in each sample was estimated at the same time by the official method. The following are the results:

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* The sand is used to prevent contact between the oxalate and the calcium carbide, which might cause loss of acetylene at ordinary temperatures. See paper on ammonium oxalate.

Nature of Sample.	Total Volatile Matter.	Moisture.	Acetone.
	Per Cent.	Per Cent.	Per Cent.
Cordite M.D.	0·41	0·29	0·12
Cordite M.D....	0·72	0·24	0·48
Cordite returned from India	0·36	0·23	0·13
First sample after softening with acetone for grinding...	5·39	0·60	4·79
Cordite Mk. 1, recent, size 20	0·27	0·11	0·16
Cordite Mk. 1, size 20, old ...	0·40	0·10	0·30
Cordite M.D., recent ...	1·04	0·34	0·70
Cordite M.D., old	0·80	0·44	0·36

In order to test the accuracy of these results the following experiments were made:

(*a*) Two samples were examined, each in duplicate, in order to see whether the results were concordant.

(*b*) A sample was taken and the moisture estimated as before; the experiment was then repeated with another portion, to which a drop of acetone had been added after weighing, to find out the effect, if any, of the presence of this body.

(*c*) A portion, previously dried, was weighed, exposed to moist air and again weighed, and the moisture so absorbed estimated as before.

The results were as follows:

(*a*)

	Sample I.	Sample II.
First experiment	0·148 per cent.	0·114 per cent.
Second experiment	0·146 ,,	0·118 ,,

(*b*)

Sample III.

Direct	0·416 per cent.
+ acetone	0·419 ,,

(*c*)

Sample IV.

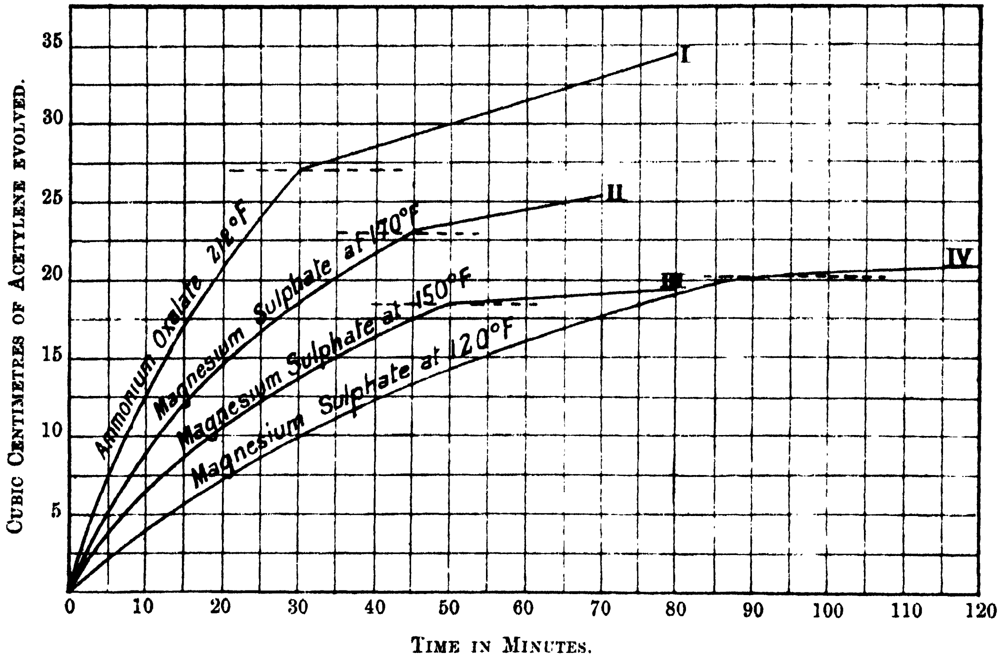
Water absorbed ...	0·345 per cent.
Water found	0·344 ,,

The method has also been applied to other substances, and found very useful in the case of a mixture containing a volatile constituent, such as naphthalene or camphor.

For instance, about 0·25 gram of naphthalene was taken and 0·0255 gram of water added; the moisture estimated by the above method came to 0·026 gram.

Again, in an explosive containing such ingredients as ammonium oxalate or magnesium sulphate, the total moisture can be rapidly estimated by this method, whereas it takes a long time—several days in many cases—for these salts to lose all their water of crystallization *in vacuo*, especially in such explosives as gelignite. The water present as moisture can readily be calculated when the proportion of the salt has been estimated.

Experiments are now in progress to find out whether the method can be applied to volatile liquids.



Experiments made in yet another direction with the view of distinguishing, if possible, between water not in combination and combined water have yielded results which may prove to have some useful application. These experiments were carried out exactly as described in the paper on ammonium oxalate, except that the salt was taken in a moist condition, a known weight of water having been added, and the curve, representing the rate of loss of water at constant temperature, obtained. The results show that the proportion of uncombined water can be separated very approximately from the combined water by means of a sharp change in the steepness of the curve, which occurs directly all the uncombined water has been driven off.

It might be thought at first sight that a salt when so heated would lose added water only until all this had been driven off, and that it would only then begin to lose combined water. From the few experiments made, however, this does not appear to be the case; but, on the contrary, combined water commences to be lost at once, the loss continuing during the whole period taken for the uncombined water to the driven off. Also, this loss appears to take place at an average rate, during this period, of one-half the rate of loss of crystalline water after all uncombined water has gone.

The chart on p. 216 gives the results obtained. The black lines represent the observed volumes of acetylene, the observations being taken every five minutes and each marked by a circle. The dotted lines represent the heights at which the breaks the curves should, theoretically, have occurred, calculated from the amounts of uncombined water taken and allowing for temperature and pressure, and also making the allowance above stated for the loss of crystalline water. The rate of loss taken is one-half that indicated by the curve after the break.

	Weight of Uncombined Water taken.	Weight calculated from Curves.
Curve I.	0·0080	0·0080
,, II.	0·0325	0·0323
,, III.	0·0275	0·0274
,, IV.	0·0325	0·0325

Discussion.

The President (Mr. Bevan) having invited discussion,

Mr. Guttmann said that the differentiation of the various volatile matters in cordite was not so unimportant as might at first be thought. Having regard to the important rôle that any volatilizable matter in a powder must play as far as ballistic results were concerned, it would be easily realized what an enormous difference there was between water and acetone. When it was remembered that ½ per cent. of moisture might make an explosive absolutely unexplodable, while with ¼ per cent. it might still be explosive, and having regard to the narrow margin allowed by official specifications and shooting tests, the great influence of even ¼ per cent. of moisture would be readily understood. He remembered a paper by Mr. A. Marshall in the *Journal of the Society of Chemical Industry*, in which special means were described for condensing the nitro-glycerin, which would also be evaporated in the process. Mr. Dupré had not alluded to this, but presumably, at the temperature to which the cordite was heated, a good deal of nitro-glycerin would come off, and one wondered what happened to it. Was it included with the acetone? If so, these results would need bringing into order by some further work, and if Mr. Dupré could succeed in differentiating between the nitro-glycerin and the acetone—either by applying Mr. Marshall's process or in some other way—he would make his method complete.

Mr. A. Marshall said that the first attempt to find a satisfactory method of determining the total volatile matter in cordite was made by a cousin of Mr. Dupré at Waltham Abbey. The details of the method were not altogether satisfactory, but further experiments which he (Mr. Marshall) had carried out at Waltham Abbey had resulted in its improvement. The method brought forward by Mr. Dupré advanced the question a stage further, as it enabled one to distinguish the water from the acetone or other volatile solvent used in manufacturing. Mr. Dupré's results brought out the interesting point that in cordite of uniform composition the amount of water was almost constant. Dr. Robertson, at Waltham Abbey, had by an entirely different method obtained similar results. The question arose whether that was really the proportion of water in the cordite as used for firing, or whether it might not depend upon loss or gain of moisture from the atmosphere during the necessary process of grinding the cordite to powder. He had found that ground dried cordite exposed to the air absorbed just about the same amount of moisture in a few minutes, and as the preparation of the sample took at least several minutes, he thought that the constancy of the moisture might perhaps be accounted for in that way.

Mr. DUPRÉ, in reply, said that no doubt a certain amount of nitro-glycerin was volatilized; but he had heated some carefully-dried cordite in the apparatus for ten or fifteen minutes without the evolution of any measurable amount of gas, and he had therefore concluded that whatever nitro-glycerin might be driven off was not decomposed in its passage through the carbide, but was condensed again probably at some part of the tube between the cordite and the nitrometer, and did not affect the volume of gas in the latter. The acetone distilled off would also be condensed, and a small allowance was made for it in the vapour tension. He might say that the method he had used was not intended in the least to supersede the official method of estimating the volatile matter, but it had struck him that it might be useful if the moisture and acetone could be separated. It was certainly possible that Mr. Marshall's explanation with regard to moisture might be correct, but in that case one would have to assume that 0·26 per cent. was the most that the cordite would ever take up, which from some of the results quoted does not appear to be the case.

* * * * *

THE ESTIMATION OF FAT IN HOMOGENIZED MILK.

BY H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, May 2, 1906.*)

HOMOGENIZED milk, or milk in which the fat is broken up into very small globules, not usually exceeding 0·001 mm. in diameter, is now an article of commerce. It is obtained by forcing milk under a pressure of from 200 to 400 atmospheres through very small openings; there are several machines made for this purpose, differing from each other chiefly in the method of formation of the small openings. The cream in homogenized milk no longer rises freely, but remains to a very great extent distributed throughout the whole volume.

The process is chiefly applied to sterilized milk, which is often required to stand for a long time before being used, and from which it is an undesirable thing for the cream to separate; but milk which has to stand—*e.g.*, on refreshment-bar counters—is also sometimes homogenized. It is therefore quite possible that some of the samples of milk taken under the Sale of Food and Drugs Act may be homogenized. As it appeared more than probable that the estimation of fat, especially by dry extraction processes, would be rendered more difficult by the fine state of division of the fat, I have made a few comparative experiments by different methods.

The methods chosen were those in common use by public analysts, together with the Kieselguhr method. In the Gottlieb and Werner-Schmid methods the original plan of taking an aliquot portion was not followed, but the whole of the fat was dissolved out by repeated extraction. The Adams method was carried out by extracting a blank coil into a flask tared against the flask in which the fat was weighed. In the other methods the flask was, after weighing, washed out with petroleum ether, and weighed again with any residue of substance not soluble in petroleum ether. Very slight residues were almost always obtained, but so small, if due care had been employed, that the percentage of fat was barely affected. The following are the results:

Gottlieb.	Kieselguhr.	Werner-Schmid.	Adams.	Gerber.
3·79	3·80	3·81	3·58	3·78
3·70	3·70	3·74	3·52	3·70
3·66	3·66	3·67	3·53	3·70
3·46	3·46	3·45	3·32	3·47
3·86	3·87	3·88	3·66	3·89
3·93	3·94	3·98	3·81	3·95

It is seen that though the Werner-Schmid method has a tendency to be a trifle higher than the others, the first three methods are in almost perfect agreement, while the Adams method is always low. The Gerber method also gives good results with homogenized milk.

Microscopic examination shows that the fibres of the paper used for the Adams coils are very many times larger than the fat globules, while the diatoms of Kieselguhr have openings which are much smaller. Though the Adams coil provides sufficient surface to insure that the thickness of the film of milk solids is of the some order as the size of the fat globules in ordinary milk, and thereby exposes them to the solvent action of ether, it fails to do so when homogenized milk is extracted. The film is of sufficient thickness for some of the fat globules in homogenized milk to be completely surrounded with a layer of solids insoluble in ether. With kieselguhr there is a much larger surface, and the layer of milk solids is not thick enough to completely surround the globules.

The advent of homogenized milk renders it necessary to remove the Adams method from the position it has so long occupied as a standard method. In my laboratory it has for some time been discarded in favour of the Gottlieb method, which for ease and accuracy appears to be the best method we have, and I have little doubt that it will be in the future almost universally used.

* * * * *

THE ANALYSIS OF DRIED MILK.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, May* 2, 1906.)

Milk which has been evaporated to dryness is sold as dried milk or milk-powder. The methods of drying milk vary. The milk in one process is passed over polished steel rollers heated to a temperature above that of boiling water, and the film from which most of the water has been evaporated is taken off by knives set in a suitable position; in another process the milk is condensed in a vacuum pan to one-third or one-fourth of its bulk, and is then dried in a vacuum chamber in thin layers; in a third process the milk is, after a preliminary concentration, dried on hot rollers *in vacuo;* and there are other methods which have not come into extensive use.

The analysis of these milks requires a few slight modifications of the methods used for ordinary milk. The sample should be ground and well mixed to secure uniformity. *Moisture* is estimated by drying about 1 gram in the water-bath.

Fat cannot be estimated by direct extraction, as the results are always low. The Werner-Schmid method is suitable. If there is no sugar except milk-sugar, the fat, after drying, should be dissolved in petroleum ether, and any residue weighed and subtracted from the total weight. In the presence of much of any other sugar, it is preferable to mix the ethereal solution with an equal bulk of petroleum ether, and shake out with water rendered slightly alkaline with ammonia before the solution is evaporated.

Milk-sugar may be easily and quickly estimated polarimetrically; 10 grams of milk-powder are ground up in a mortar with sufficient hot water to make it into a paste, which is gradually thinned with hot water, and the solution made up to 100 c.c.; a little ammonia may be added if the milk-powder does not all go into solution. Unless this procedure is followed, incomplete extraction of the sugar may result. The usual method is then followed.

Cane-sugar may be conveniently estimated by the method described by Harrison (ANALYST, xxx., 123).

Proteids are calculated from the total nitrogen by Kjeldahl's method by the factor 6·87.

Ash, Lime, and Phosphoric Acid as usual.

Acidity is estimated by grinding up about 1 to 2 grams with hot water, and titrating with $\frac{N}{10}$ alkali, using phenolphthalein as indicator.

The table below gives the analysis of seven samples:

TABLE I.—COMPOSITION OF DRIED MILK.

	1	2	3	4	5	6	7
Moisture	6·39	4·92	3·30	3·55	4·74	5·15	6·03
Fat	27·35	27·98	23·97	2·55	29·16	19·90	25·60
Milk-sugar	31·42	34·16	37·32	45·60	32·24	34·96	32·83
Cane-sugar	—	1·25	1·53	2·80	—	—	2·00
Proteids	27·48	24·59	26·38	35·45	26·66	31·10	23·84
Ash	6·00	6·24	6·19	7·89	5·63	7·11	6·44
Total	98·64	99·14	98·69	97·84	98·43	98·22	96·74
Water of hydration ...	1·65	1·80	1·96	2·40	1·70	1·84	1·73
Total	100·29	100·94	100·65	100·24	100·13	100·06	98·47
Change of temperature on mixing with water ...	-0·2°	±0·0°	-0·2°	-0·4°	-0·2°	—	-0·3°

It is noticed that none of the analyses add up to 100 per cent., but are considerably low; the milk-sugar has been calculated as anhydrous sugar, and here, I think, is the reason for the deficiency.

On shaking the solid residue obtained by drying milk on the water-bath, in which the milk-sugar certainly exists as anhydrous sugar, with water a rise of temperature always takes place; anhydrous milk-sugar mixed with water always causes a rise of temperature, whilst hydrated milk-sugar causes a fall of 0·55° if more than can be at once dissolved is added. The milk-powders examined, with one exception (No. 2), all caused a fall of temperature, and it is seen that the addition of the water of hydration to the total gives figures which are but slightly in excess of 100 per cent.; both the change of temperature and the slight excess over 100 per cent. indicate that the bulk of the milk-sugar, though not all, exists as hydrated sugar. Sample No. 2 differed in appearance from the others, being a heavy powder, instead of being light and flaky, and had doubtless been more dried, and probably contains a considerable proportion of anhydrous sugar; it is noticed that the addition of the water of hydration would make the total nearly 101 per cent. Sample No. 7 gives a low total, which I believe is accounted for by the presence of invert sugar.

It will be noticed that samples 2, 3, 4 and 7 contain small quantities of cane-sugar; this in sample 2 was admittedly added in the form of saccharate of lime, and was certainly so added, judging from the analytical figures, in No. 7. This is used to render the dried milk more soluble, and additions of sodium phosphate and other alkaline substances are made with the same object.

In Table II. the composition of the original milks, on the assumption that they contain 9·0 per cent. of solids-not-fat, are given.

TABLE II.—COMPOSITION OF ORIGINAL MILKS.

Fat	3·79	3·88	3·09	0·26	4·07	2·45	3·65
Milk-sugar	4·36	4·73	4·81	4·62	4·50	4·30	4·68
Proteids	3·81	3·41	3·40	3·58	3·71	3·82	3·40
Ash	0·83	0·87	0·80	0·80	0·79	0·87	0·92
CaO	0·19	0·21	0·17	0·17	0·17	0·19	0·27
P_2O_5	0·23	0·24	0·23	0·23	0·23	0·29	0·24
Acidity	8·4°	13·2°	16·8°	16·5°	19·6°	—	11·4°

From this table it is seen that No. 4 is made from separated milk, and No. 6 from milk deprived of a portion of its cream. The milk used to prepare No. 3 is only just above the Government standard. The normal percentages of lime and phosphoric anhydride in milk are 0·17 per cent. and 0·23 per cent. respectively, but vary somewhat with the proteids, and the normal acidity is not far from 20°. From a consideration of the results, it would appear that Nos. 2 and 7 have received an addition of saccharate of lime; and No. 6 has received an addition of a phosphate. Nos. 3 and 4 contain cane-sugar, but there is no evidence of the addition of saccharate of lime. No. 1 has probably received an addition of sodium carbonate, as the lime is not high enough, considering the high proteids, to indicate an addition of this substance; and No. 5 appears to have received no addition whatever.

Discussion.

The **President** (Mr. Bevan), in inviting discussion, said that he could not quite understand how it was that, in the case of homogenized milk, the Adams process gave low results as compared with the Werner-Schmid and other methods. He should have thought that the breaking-up of the fat globules would have caused them to be more readily soluble. He should be interested to hear by what process the samples of dried milk referred to were made.

Mr. W. **Partridge** asked whether any unusual difficulty had been found in extracting the fat with ether in the case of homogenized milk. He had had occasion to examine some samples of condensed homogenized milk in which the degree of homogenization was stated to be very high, and, using the Werner-Schmid process, his results were similar to those of a Norwegian analyst who had examined the same milks; but a London analyst who had also examined them, by Bell's process, had obtained results which were regularly 1 per cent. lower.

Mr. **Hehner** said that he had made some time ago a few comparative experiments with regard to fat extraction in the case of dried milk prepared by the Hatmaker process, and had found in every case that a larger proportion of fat was obtained by extracting the powder itself with ether than by any other means. When plaster of Paris and other like materials were used lower results were invariably obtained. In the Hatmaker process, as in all others, alkali was added in the form of sodium carbonate, which would account for the somewhat high ash. He wondered why Mr. Richmond was so anxious that his analyses should add up to 100, seeing that there must necessarily be a number of undetermined constituents—for instance, there would be about $\frac{1}{10}$ per cent. of citric acid in the milk, which, of course, would represent about $\frac{8}{10}$ per cent. in the dried product. He thought that the reason why, in the case of homogenized milk, the fat was not well extracted from paper was not quite satisfactorily explained by the photographs shown. When milk was mixed with an absorbent it evaporated, and the fat remained more or less adhering to the surface presented to it. It could not surely make much difference whether the absorbent was in small or large particles. The question must be whether the surface on which the fat was, so to speak, plastered was large enough to subdivide it completely, and he could not see why the surface presented by one material should be less favourable than that presented by another.

Mr. M. **Wynter Blyth** said that, from some experiments which he had made with certain foods for infants, he could confirm what Mr. Richmond had said as to the difficulty of extracting the whole of the fat by the Adams process. In one case he could only extract about 2 per cent. of fat, whereas a modification of the Werner-Schmid method, which he had used on Mr. Richmond's suggestion, yielded 8·24 per cent. of fat. The low results given by the Adams process in such cases were explicable by the fact that, especially with malted foods, a dry mass of sugar was obtained which was very difficult to deal with. In the case, however, of milk in which the fat globules had been broken up, he quite agreed with Mr. Hehner that the reason why the fat could not all be got out was difficult to understand.

Dr. LEATHER inquired how long the extraction with ether was carried on.

Mr. E. R. BOLTON asked whether the fat extraction was carried out in a Soxhlet apparatus or in one of those extractors in which the solvent dripped down and trickled straight through. He had found that in the case of oil-cakes all the fat was extracted much more quickly in such an extractor than in the Soxhlet apparatus.

Mr. WYNTER BLYTH said that in his case an ordinary Soxhlet apparatus was used, and the extraction carried on for five hours.

Mr. RICHMOND, in reply, said that the milk-powders referred to were obtained by the various processes described in the paper, but he did not know in all cases which process had been used. Some of the samples were very finely ground, and had been dried to a greater extent, and probably contained a larger amount of anhydrous milk-sugar. It seemed to him that the photographs went a long way towards explaining the lowness of the Adams results. It must be borne in mind that the photographs (not reproduced) were on different scales. Had they been on the same scale the enormous difference in the total surfaces exposed to the milk would have been more apparent. In the case of kieselguhr the layer was of the same order of thickness as the fat globules, and consequently they were all exposed to the action of the ether. Probably, also, the grinding-up of the milk and kieselguhr had some effect. In the determinations by the Adams method extraction was carried on for at least five hours in a Soxhlet apparatus. In some cases extraction was carried on for so long as sixteen hours, but the full proportion of fat was still not obtained. He had not found any difficulty with the Werner-Schmid process. He never made more than four extractions, and at the fourth there was generally practically no fat left to take out. He had compared various methods of extracting, and had found four extractions at the outside to be sufficient. This applied also to homogenized condensed milk.

Mr. PARTRIDGE asked how much ether Mr. Richmond used for each extraction. He himself used about 15 or 20 c.c.

Mr. RICHMOND said that he used perhaps a little more, but not very much. In the case of homogenized milk he thought that the Gottlieb method had advantages over the Werner-Schmid. It took much less trouble, and the fat came out more cleanly and easily. With regard to the addition of alkali, he thought that sodium phosphate was used quite as often as sodium carbonate.

Mr. HEHNER said that he had been consulted as regards the manufacture of dried milk both on the Continent and in America, and had never known any alkali but sodium carbonate to be used.

Mr. RICHMOND said that he knew that sodium phosphate had been used in England. With regard to the milk-sugar, having obtained distinct evidence from the change in temperature on mixing with water that there was some water of hydration, he thought it only right to include this. He did not, of course, suggest that these analyses ought necessarily to add up exactly to 100. As Mr. Hehner had mentioned, there was citric acid present, which, however, was partly compensated for by the fact that a portion of the phosphoric acid was included both in the casein and in the ash. Analyses of dried milk should, however, not be 1½ to 2½ per cent. low.

Mr. Fairley asked what, if the Adams process failed when the milk was spread over a certain surface, was to prevent its being spread over a larger surface by dilution if necessary, and whether, in the Werner-Schmid determinations, any attempt had been made to find out whether any fixed residue was contained in the fat, because, after all, these discrepancies were minute, and a very small amount of impalpable mineral matter in the fat might make all the difference.

Mr. Richmond said that there was really nothing to prevent the milk being spread over a larger surface. The fat in the Werner-Schmid determinations was tested to see if it contained anything insoluble in petroleum ether, but was not ignited to see if there was any non-volatile matter.

Note.—In the *Journal of Analytical Chemistry*, vol. iv., part i., I stated : "From the data given by Adams (Analyst, x., 46), I calculate that on paper the thickness of the film of milk solids is less than 0·0002 inch, and from my own measurements of kieselguhr, I conclude that the film dried up on this medium is about 0·0000005 inch. . . ."] H. D. R.

* * * * *

NOTE ON A RECENT PAPER BY R. STEINEGGER ON THE "ALDEHYDE FIGURE" OF MILK.

By H. DROOP RICHMOND, F.I.C., and E. H. MILLER.

(*Read at the Meeting, May* 2, 1906.)

We have been induced to refer to this paper by the fact that the method (Analyst, xxxi., 46) proposed is likely to be of considerable use.

The author first shows that formalin added to milk hinders and finally prevents curdling by rennet. Next he discusses the increase of acidity on the addition of formalin pointed out by Hanne and Hesse, and shows that the acidity increases with the amount of formalin used and attains a maximum when about 5 per cent. (at least 1·8 per cent. of formaldehyde) is added, whether the addition is made all at once or in successive amounts.

He then gives the variations in the milk of *single cows*, and it is not pointed out in the abstract that the maxima and minima given are in the milk of single cows. Next he shows that the increase of acidity is not due to the action of a ferment causing oxidation to formic acid, as had been suggested by Hesse, and that it remains constant whether rennet is added to the milk or not. In addition to giving figures that show that the aldehyde figure is proportional to the total nitrogen in the milk, and that the total nitrogen (and therefore the percentage of proteids) can be calculated from the aldehyde figure, he gives some experiments with tyrosine and leucine, which show that the acidity is produced by the conversion of the amino groups into methylene-amino groups by condensation, with the consequent conversion of the amphoteric nature of the amino-acid into an acid one.

We think this paper important because—

1. The method appears to give an approximate estimation of the amino-nitrogen of the proteids.
2. It gives an indirect but very easy method for proteid estimation.
3. It adds another figure to those which can be easily determined in milk analysis, and thus will yield corroborative evidence.

Our first experiments were made with a view to see if we could obtain definite evidence of the combination with the amino-groups, and experimented with whey produced by the action of rennet and acid respectively.

Our results were:

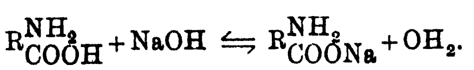
	Treated with Benzene-diazonium Chloride.	Diazotized and combined with β-naphthol.
Acid whey	Dark red colour	Bright yellow precipitate
Acid whey + CH_2O ...	Dark yellow colour	No reaction
Rennet whey	Bright red colour	Light yellow colour
Rennet whey + CH_2O ...	Light yellow colour	No reaction

In each case the wheys effervesced in the cold on treatment with nitrous acid, whilst after treatment with formaldehyde there was no gas given off in the cold. The amount of gas given off from the rennet whey corresponded to 0·013 per cent. of amino-nitrogen, while the increase of acidity corresponded to about 0·01 per cent. of amino-nitrogen on the assumption that each molecule of NH_2 corresponded to one equivalent of acidity. These results show the presence of both aromatic and fatty amino groups in milk which condense with formaldehyde.

We next experimented on a casein solution containing 0·2596 gram nitrogen by Kjeldahl per 100 c.c.; this gave an aldehyde figure of 9·8 c.c. $\frac{N}{10}$ soda per 100 c.c., and combined with 0·0394 gram CH_2O per 100 c.c., which is equivalent to 13·2 c.c. $\frac{N}{10}$ alkali. Ten c.c. of this solution gave 0·1601 gram casein by precipitation with acetic acid, and 0·1608 gram after the addition of formaldehyde.

These results show that the "aldehyde figure" is not a complete measure of the amino-groups nor of the formaldehyde condensed.

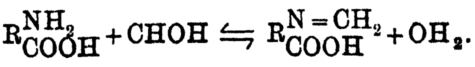
Proteids certainly contain both amino and carboxyl groups, and when alkali is added (*e.g.*, soda) the following reaction takes place:



$$R^{NH_2}_{COOH} + NaOH \leftrightharpoons R^{NH_2}_{COONa} + OH_2. \qquad (1).$$

The point of neutrality to phenolphthalein is, on account of the presence of the basic NH_2 group, much nearer to the acid than to the sodium salt.

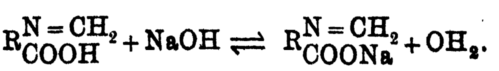
The action of formaldehyde is probably—



$$R^{NH_2}_{COOH} + CHOH \leftrightharpoons R^{N=CH_2}_{COOH} + OH_2. \qquad (2),$$

and the reaction in the presence of a large excess of formaldehyde is probably complete and very rapid.

The methylene-amino acid reacts with alkali thus:



$$R^{N=CH_2}_{COOH} + NaOH \rightleftharpoons R^{N=CH_2}_{COONa} + OH_2. \qquad (3),$$

and the point of neutrality is probably very close to the salt on account of the feeble basicity of the methylene-amino group. The "aldehyde figure" measures the difference between the points of neutrality of the two reversible reactions (1) and (3).

It is therefore by no means certain that all alkalies will give the same "aldehyde figure." We have measured in four samples of milk the "aldehyde figure" with soda and strontia respectively. The results are expressed as c.c. N alkali per litre.

Aldehyde Figures with

	Soda.	Strontia.
1.	17·4	20·2
2.	20·1	21·6
3.	18·8	20·2
4.	21·8	22·6

The strontia "aldehyde figure" is always higher than the soda figure, and the mean ratio is 1·1.

We also find in mixed milk that soda gives a mean "aldehyde figure" of 18·4, while strontia yields 20·2, also in the ratio of 1·1.

Steinegger, who used the Soxhlet-Henkel method of determining acidity, probably used soda as his alkali. We find that with soda 1 c.c. $\frac{N}{10}$ alkali corresponds to 0·0265 gram casein nitrogen, and 0·053 gram albumin nitrogen. Steinegger finds that on the average 1 c.c. $\frac{N}{10}$ alkali corresponds to 0·0303 gram of the nitrogen of milk (1 degree Soxhlet-Henkel = 2·5 c.c. $\frac{N}{10}$ alkali per 100 c.c. = 0·0758 gram N per 100 c.c.), whilst, taking the mean proportion of casein to albumin to be 7 : 1, we find 0·0298 gram using soda and 0·0271 gram using strontia.

As the titration is rather sharper with strontia than with soda, we prefer the use of the former solution.

The mean of 113 determinations gives 19·9 c.c. N strontia per litre of milk as the mean "aldehyde figure," with 22·6 c.c. as a maximum and 18·1 c.c. as a minimum. As showing the relative constancy in milk from a herd, we give the highest figures, the lowest figures, and a mean figure. All figures were on different dates:

	Aldehyde Figures.	Mean Solids-not-Fat.
Highest herd	21·0, 22·0, 22·0, 22·6	9·16
Mean herd	19·6, 20·2, 19·6, 19·6, 19·6	8·97
Lowest herd	18·1, 18·6, 18·1	8·61

We have also made a number of experiments on the change in the aldehyde figure when milk is acted on by micro-organisms, and on the amino groups in milk proteids. These will be discussed later.

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ANNIVERSARY DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

The anniversary dinner of the Society took place on Wednesday evening, June 13, at the Trocadero Restaurant, under the chairmanship of the President, Mr. E. J. Bevan. Among the guests present were Sir Thomas Elliott, K.C.B., Secretary of the Board of Agriculture and Fisheries; Mr. John Lithiby, C.B., Assistant Secretary of the Local Government Board; Professor Percy F. Frankland, LL.D., F.R.S., President of the Institute of Chemistry; Dr. Divers, F.R.S., President of the Society of Chemical Industry; Mr. R. A. Robinson, President of the Pharmaceutical Society; Mr. Montague Baird, President of the Institute of Brewing; Dr. T. Cooksey, Deputy Analyst to the Government of New South Wales; Sir William Ramsay, K.C.B., F.R.S.; Professor J. Millar Thomson, LL.D., F.R.S.; Professor W. A. Tilden, D.Sc., F.R.S.; Dr. Buchanan; Mr. F. W. Beck; Mr. Richard B. Pilcher, Secretary of the Institute of Chemistry; and Mr. Aubrey W. Rake.

The loyal toasts having been duly honoured, the following toasts were proposed: "Government Departments," by Sir William Ramsay, responded to by Sir Thomas Elliott and Mr. Lithiby; "The Society of Public Analysts," by Professor J. Millar Thomson, responded to by the President; "Kindred Societies," by Mr. Bertram Blount, responded to by Professor Frankland and Dr. Divers; and "The Guests," by Mr. A. Gordon Salamon, responded to by Dr. Buchanan and Mr. R. A. Robinson. A choice selection of music, tastefully rendered by a select party of glee singers, added much to the enjoyment of the evening.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Milk Substitute. R. Racine. (*Zeit. öffentl. Chem.*, 1906, xii., 167, 168.)—A preparation, sold to confectioners, under the name of artificial milk, has recently been examined by the author and found to consist essentially of sesame oil, sugar, and albumin. The results of the analysis were:

Total solids	85·90	per cent.
Ash	0·16	,,
Oil (sesame)	47·10	,,
Sugar (as invert sugar)	34·00	,,
Albumin	4·51	,,

The preparation was of a syrupy consistency, and, when mixed with water in any proportion, gave milky solutions from which the oil separated very slowly.

W. P. S.

Adulteration of Butter with Casein. R. Racine. (*Zeit. öffentl. Chem.*, 1906, xii., 169-170.)—A sample of butter was found by the author to have the following composition: Fat, 69·01 per cent.; water, 24·73 per cent.; ash, 0·77 per cent.; casein, 5·49 per cent. This form of adulteration is readily detected owing to the large quantity of coagulum obtained on melting the sample. It will also be seen that an excessive quantity of water was contained in this butter. W. P. S.

The Detection of Bleached Flour in Wheat Flour. E. Fleurent. (*Comptes Rendus*, 1906, cxlii., 180; through *Journ. Pharm. Chim.*, 1906, xxiii., 499, 500.)—The only processes of bleaching flour that have an industrial value are those in which nitrogen peroxide is used as the reagent. Pure oxygen or ozone does not affect the colour, and although ozonized air has a bleaching effect, it is only when it is simultaneously charged with nitrous products. Moreover, flours treated with ozone acquire a repulsive odour, which destroys their commercial value. The quantity of nitrous vapours in terms of nitrogen peroxide absorbed ranges from about 15 to 40 c.c. per kilogm. of flour. The nitrogen peroxide forms an addition compound with the fat, the iodine value of which is correspondingly lowered. The action of ozone, however, causes an increase in the iodine value and the formation of volatile fatty acids. The fixation of the nitrogen peroxide by the fat affords a means of differentiating bleached from normal flour. Fifty grams of the sample are extracted with petroleum spirit (benzine), the extract evaporated at a low temperature, the residue of oil dissolved in 3 c.c. of amyl alcohol, and the solution shaken with 1 c.c. of a 1 per cent. alcoholic solution of potassium hydroxide. In the case of a normal flour the resulting soap solution is of the same pale yellow colour; but when the flour has been bleached the colour changes to reddish-orange, the depth of shade depending upon the amount of nitrogen peroxide that had been fixed by the fat. The test is capable of detecting the addition of 5 per cent. of bleached flour to ordinary flour. The bleaching process has no action upon the diastase or other enzymes of the flour, but the fat becomes acid more slowly than that which has not combined with nitrogen peroxide. It is in this sense only that the bleaching process can be said to have any preservative action upon the flour. C. A. M.

A Study of the Proteids of Beef-Flesh. P. F. Trowbridge and H. S. Grindley. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 469-505).—The results of their previous work (ANALYST, 1905, 89) led the authors to the conclusion that much additional information regarding the proteids of flesh could be obtained by extracting the flesh successively with cold water, with a 10 per cent. ammonium sulphate solution, and finally with $\frac{N}{20}$ potassium hydroxide solution in the cold. An investigation on these lines was therefore carried out, together with a study of the acidity of the flesh, with the following results: The total acidity of aqueous extracts of flesh varies between comparatively wide limits, the minimum being 0·66 per cent., the maximum 1·07 per cent., and the average 0·85 per cent., calculated as lactic acid and expressed in terms of the fresh meat. Whilst the residues of flesh which are left after complete extraction with cold water are distinctly acid to litmus and to phenolphthalein, the insoluble proteids of flesh upon digestion with $\frac{N}{10}$ hydrochloric acid, at the ordinary temperature, combine with the latter, neutralizing its acid properties.

The analyses show that 13·56 per cent. of the total proteids existing in lean flesh is soluble in cold water. Of this, 90·04 per cent. is coagulable by heat from a neutral solution, 8·40 per cent. exists in the form of albumoses, and a very small quantity apparently as peptones. It is not at present supposed that the albumoses and peptones exist as such in the original flesh. During the coagulation of an aqueous extract of flesh there is an increase in the acidity of the same. Reduction of the acidity of the extract facilitates the coagulation of the proteids, the separation of the coagulable proteids being more complete with one-fourth neutralization (to phenolphthalein) of the acidity of the extract than it is on further neutralization. The partial neutralization of such extracts causes the precipitation of some proteid matter at the ordinary temperature. There is no well-defined temperature at which different coagula of flesh extracts separate. The complete removal of the proteid coagulating at any definite temperature requires long application of heat, and the lower the temperature at which a coagulum is separated, the longer the time required to effect the separation.

Of the total proteid of a cold water extract of flesh, 11·71 per cent. is separated by neutralization, 36·65 per cent. is coagulable below 50° C., 39·99 between 51° and 75° C., and 11·71 between 76° and 85° C. The chemical composition of the different fractional coagula is remarkably constant, and, judging from the results of their hydrolysis, their chemical constitutions are quite similar.

Raw flesh which has been completely freed from proteids soluble in cold water contains two classes of proteid substances—those which are soluble in 10 per cent. ammonium sulphate solution and those which are insoluble. Of the total proteids of lean flesh about 16 per cent. are insoluble in cold water, but soluble in 10 per cent. ammonium sulphate solution. The latter solution extracts from flesh at least two individual proteids or groups of proteids which differ in physical properties, and to some extent in chemical composition.

The residue left after extracting raw flesh with cold water and 10 per cent. ammonium sulphate solution is almost entirely soluble in $\frac{N}{20}$ potassium hydroxide solution, and has the same chemical composition even when separated from the solvent by different means and purified by widely different treatment.

W. P. S.

The Presence of Sodium Arsenate in Preserved Meat. A. Andouard. (*Journ. Pharm. Chim.*, 1906, xxiii., 417, 418.)—Sodium nitrate has gradually replaced potassium nitrate as a means of giving to preserved pork the rosy colour of the fresh meat. In the author's opinion, it is less injurious than nitre, but he considers that the use of any alkaline nitrate for such purposes should be prohibited, both on account of their active medicinal properties and of the risk of poisoning from the use of impure salts. Several instances of such poisoning have come under his notice, and in three cases the supposed sodium nitrates used for colouring the flesh were found to contain sodium arsenate.

C. A. M.

Determination of Fat in Cocoa. A. Kirschner. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, pp. 450, 451.)—The following manner of applying the Gottlieb process (ANALYST, 1898, 259) to the determination of fat in cocoa is described: A weighed quantity of about 1·5 grams of the cocoa and 20 c.c. of alcohol (50 per cent. by volume) are shaken together in a graduated cylinder. The latter should have a capacity of 75 or 100 c.c., be divided into divisions of 0·5 c.c., and be of such a diameter that the height between the 0 and 75 c.c. marks is not less than 30 cm.; 25 c.c. of ether are then introduced, and the mixture shaken for fifteen minutes, when 25 c.c. of petroleum spirit are added and mixed by carefully inverting the cylinder a few times. Care must be taken not to form an emulsion. The cylinder is now set aside for one hour to allow the solvent to separate, 45 c.c. of the ethereal layer being then withdrawn, evaporated, and the residue of fat weighed. As the presence of the cocoa prevents the total volume of the solvent being read off, a blank experiment is made, in which the above-mentioned quantities are used, but the cocoa omitted, and the volume thus ascertained. To the total volume found in this manner is added the weight of fat obtained, 1 c.c. being added for each gram. The amount of fat in the cocoa is then found by a simple calculation. From the results given the process appears to be trustworthy. W. P. S.

The Determination of Foreign Substances in Cocoa and Chocolate. F. Bordas and Touplain. (*Comptes Rendus*, 1906, xlii., 794; through *Journ. Pharm. Chim.*, 1906, xxiii., 502, 503.)—Foreign substances are usually searched for with the microscope in the insoluble residue left after extraction of the fat with ether and of the sugar with water. To simplify the process, the authors have devised a mechanical method of separating the different substances by treating the residue successively with mixtures of carbon tetrachloride and petroleum spirit (benzine), ranging in specific gravity from 1·340 to 1·600. Preliminary experiments have shown that the different substances that may be in the insoluble residue behave as follows on treatment with the different mixtures:

Specific Gravity.	Insoluble Residue of—	Behaviour.
1·340	Arachis cake	Sinks
1·435	,,	Floats
1·400	Cocoa germs	Sink
1·440	,,	Float
1·440	Pure cocoa	Sinks
1·500	,,	Floats
1·500	Husks	Sink
1·530	,,	Float
1·510	Potato starch	Sinks
1·525	,,	Floats
1·600	Mineral matter	Sinks

C. A. M.

A Reaction of Theobromine. G. Gérard. (*Journ. Pharm. Chim.*, 1906, xxiii., 476, 477.)—A small quantity (0·05 gram) of the alkaloid is mixed with 3 c.c. of water and 6 c.c. of sodium hydroxide solution (*lessive des savonniers*), and the tube allowed to stand until the liquid becomes clear, after which 1 c.c. of ammonium hydroxide and 10 c.c. of a 10 per cent. solution of silver nitrate are introduced. The mixture on shaking forms a colourless transparent mass, which becomes fluid at 60° C., and sets to a *transparent* jelly when cooled again. If the heating be done too rapidly, too concentrated solutions of the alkaloid yield opaque jellies. In the author's opinion, the reaction is probably due to the formation of a silver precipitate of theobromine in a gelatinous state similar to that of precipitated silica. Caffeine does not give the reaction. The test is very sensitive, and it is possible to obtain evident solidification with 0·01 gram of theobromine in 10 c.c. of liquid. When once formed, the jelly can be kept for several weeks without undergoing any alteration.

C. A. M.

Detection of Sugar in Mace. E. Spaeth. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, pp. 447-450.)—Notwithstanding the fact that mace contains naturally a small quantity of sugars (*cf.* ANALYST, 1905, 207), the presence of added sugar may be detected by the following test, described by the author some years ago (1896): Ten grams of the sample, previously freed from fat by means of petroleum spirit, are shaken with chloroform and allowed to settle. After decanting the greater part of the latter, the last few c.c., together with the sediment, are evaporated on the water-bath, and the residue dissolved in a little warm water. The solution is clarified by the addition of lead acetate and aluminium hydroxide (the latter alone is usually sufficient), diluted to 50 c.c. with water, filtered, and the filtrate examined by the polarimeter. When thus treated pure samples of mace yield no polarizing substances, and, consequently, the presence of added sugar is readily detected. W. P. S.

The Ash of Pepper and Cinnamon. H. Lührig and R. Thamm. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 129-134.)—The following results were obtained on the analysis of specimens of Malabar, Tellicherri, and Singapore peppercorns, 9 samples in all being examined. The figures represent percentages on the sand-free dry substance. The amount of sand present varied from 0·040 to 0·265 per cent.:

	ASH.			ALKALINITY OF ASH: C.C. N ACID PER 100 GRAMS OF PEPPER.		
	Total.	Water-soluble.	Water-insoluble.	Total.	Water-soluble.	Water-insoluble.
Average ...	5·02	3·19	1·83	52·4	26·2	26·1
Highest ...	5·60	3·57	3·14	54·9	28·6	38·3
Lowest ...	4·67	2·46	1·49	49·1	15·7	20·5

Eight samples of Ceylon cinnamon gave the following results:

Average ...	5·28	1·56	4·72	92·2	14·7	77·5
Highest ...	5·96	1·84	4·32	104·5	18·1	88·6
Lowest ...	4·30	1·26	3·00	72·9	11·0	70·5

Two samples of cassia bark—a common adulterant of cinnamon—gave results as under:

1.	2·36	0·90	1·46	34·5	5·95	28·6
2.	2·37	0·96	1·41	35·6	6·14	29·4

For the determination of the soluble alkalinity of the ash, the latter, after being weighed, was washed into a 100 c.c. flask with about 50 c.c. of water, boiled for ten minutes, then cooled and made up to the mark. The solution was filtered, and 50 c.c. of the filtrate boiled with the addition of an excess of ½ N acid. The excess of the latter was then titrated back with ½ N alkali solution. The whole of the contents of the 100 c.c. flask were now brought on to the filter, washed with a large volume of boiling water, and the insoluble ash determined as usual. The samples of cinnamon contained very little sand—viz., from 0·02 to 0·08 per cent. W. P. S.

The Detection of Soap-Wort in Mineral Waters. J. Vamvakas. (*Ann. de Chim. anal.*, 1906, vol. 77, 161-163.)—A decoction of soap-wort root (*Saponaria officinalis*) is a common addition to mineral waters, and various preparations containing it are used to produce frothing when the bottle is opened. One of these, of English origin, examined by the author, was sold under the name of "Liqueur Lautain." It was a brown liquid with an odour of banana, an acid reaction, and a density of 1·140 at 15° C. It consisted, in the main, of an aqueous extract of soap-wort root, and contained 1·6 per cent. of sugar. When diluted with water, boiled, cooled, and treated with a few drops of Nessler's reagent, it gave a bright yellow to deep orange-coloured precipitate, which became greenish-gray after a few hours, and finally leaden-gray. A similar reaction was obtained with decoctions of soap-wort root and with mineral waters containing it. If the liquids were boiled after the addition of the Nessler's reagent, a gray precipitate, changing immediately to blackish-gray and greenish-gray, was formed. The addition of a few drops of nitric acid or of a concentrated solution of tartaric acid prevented the formation of any precipitate by the reagent. The following method of applying the test to lemonade and other mineral waters is recommended: 100 c.c. of the liquid are boiled to expel the carbonic acid and the free ammonia in the water, diluted to the original volume again, and treated with lead subacetate. The white precipitate is filtered off, suspended in water, and decomposed with a current of hydrogen sulphide, abundant frothing being produced if the original liquid contained soap-wort extract. The liquid is now filtered, and the filtrate and washings boiled until quite free from hydrogen sulphide, cooled, and divided into three parts. One of these is tested with Nessler's reagent and allowed to stand, the second boiled with the reagent, and the third tested with a few drops of a concentrated solution of tartaric acid in the hot as well as in the cold solution. If the drink contained soap-wort, a yellow precipitate will be obtained in the first case, a greenish or grayish-black precipitate in the second, and no precipitate at all in the third. The reaction is not applicable to sparkling wines or to beer.

C. A. M.

The Acids occurring in Raspberry Juice. R. Kayser. (*Zeit. öffentl. Chem.*, 1906, xii., 155, 156.)—The following organic acids were detected and their quantity determined in (1) a sample of raspberry syrup, and (2) raspberry juice preserved with alcohol. The figures express grams per 100 c.c. after allowing for the amounts of sugar and alcohol respectively present:

	1.	2.
Tartaric acid	0·220	0·180
Citric acid	0·756	0·655
Malic acid (by difference)	0·220	0·300
Volatile acid (as acetic)	0·060	0·045

W. P. S.

Composition of Raspberry Juice. Hefelmann, P. Mauz and F. Muller. (*Zeit. öffentl. Chem.*, 1906, xii., 141-155.)—The average results of the analyses of fifty samples of 1905 raspberry juice were found to be as follows:

	Total Solids. Per Cent.	Ash. Per Cent.	Alkalinity of Ash. Cc. $\frac{N}{1}$ Acid.
Average	4·09	0·430	5·61
Maximum	4·94	0·505	6·90
Minimum	0·89	0·312	4·50

These results are in agreement with those found for other years.

W. P. S.

The Composition of Tamarind Pulp. O. Remeaud. (*Journ. Pharm. Chim.*, 1906, xxiii., 424-429).—The following percentage results were obtained in the analysis of pulp prepared from the fruit by the author and of two commercial products:

	Dry Extract.	Water and Substances Volatile at 100° C.	Ash.	Substance Insoluble in Water.	Total Acids, as Tartaric Acid.	Free Tartaric Acid.	Potassium Bitartrate.	Phosphoric Acid, P_2O_5.	Other Acids, as Tartaric Acid.	Invert Sugar.	Saccharose.	Pectins.
Prepared pulp ...	62·83	37·17	2·82	6·25	11·73	5·71	6·05	0·25	3·60	42·31	0·67	0·35
Commercial pulp, purified ...	73·04	26·96	3·26	7·08	15·34	7·03	7·34	0·37	5·38	34·28	0·46	—
Commercial pulp, crude, without seeds	73·03	26·96	3·19	12·35	15·89	7·36	6·57	0·27	5·91	32·00	—	1·38

The prepared pulp differed from the commercial products in containing less total acids, somewhat less potassium bitartrate, and much more invert sugar. The fruit from which it was obtained contained 29·31 per cent. of pulp and 37·07 per cent. of seeds.

C. A. M.

Composition of Lemon Juice. H. Lührig. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, vol. 11, pp. 441-445).—The following results were obtained on the examination of ten samples of lemon juice pressed from the fruit in the author's laboratory, the figures expressing grams per 100 c.c. of the fresh juice, an allowance having been made for a quantity of alcohol added for preservative and clarifying purposes: Total solids, 10·181; citric acid (anhydrous), 7·586; ash, 0·3643; nitrogen, 0·059; invert sugar, 1·572; glycerol, 0·220; phosphoric acid, 0·0232; alkalinity of the ash, 4·99 c.c. N acid.

W. P. S.

The Detection of Camphene in Essential Oils. L. Balbiano. (*Reale Accad. dei Lincei*; through *Journ. Pharm. Chim.*, 1906, xxiii., 397.)—Camphene combines with mercuric acetate to form a crystalline addition compound, $C_{10}H_{16}OHg(C_2H_3O_2)_2$, which is insoluble in water, alcohol, and ether, and can be decomposed by a current of hydrogen sulphide with liberation of the camphene. Pinene is oxidized by the reagent, but does not yield a crystalline derivative. In applying the test, the essential oil is mixed with its own volume of benzene, and shaken with a sufficient quantity of a 25 per cent. aqueous solution of mercuric acetate (2 molecules of mercuric acetate to 1 molecule of camphene). The crystalline precipitate formed after thirty days' contact is readily purified owing to its insolubility in most neutral solvents, and the camphene can then be recovered by means of hydrogen sulphide. C. A. M.

The Lemon-scented Ironbark and its Essential Oil. R. T. Baker and H. G. Smith. (*Pharm. Journ.*, 1906, vol. 76, pp. 571, 572.)—This small tree (*Eucalyptus staigeriana*) which is only at present found in Queensland, is a species with distinct characteristics and is not easily confounded with any other eucalyptus. The leaves examined by the authors yielded 2·484 per cent. of essential oil, having a light lemon colour and an odour of citral and geraniol. The rotation of the oil in a 100 mm. tube was − 43·1; the specific gravity at 16° C. was 0·8708, and the refractive index at the same temperature 1·4871. The approximate composition of the oil was: Limonene, 60·00; geraniol, 12·72; geranyl acetate, 8·32; citral, 16·00; undetermined, 2·96 per cent. From the physical properties of the fractions obtained on distillation, it is seen that nearly the whole of the terpene present was lævo-limonene. W. P. S.

Peru Balsam Oil. Haensel. (*Haensel's Report*, 1906; through *Pharm. Journ.*, 1906, vol. 76, p. 642.)—Genuine balsam contains about 30 per cent. of essential oil which is dark yellow in colour, has a slight dextro rotation, and a specific gravity of 1·083 at 25° C. The amount of cinnamein in this oil is 84·4 per cent.

W. P. S.

Determination of Camphor in Camphor Liniment. J. Lothian. (*Pharm. Journ.*, 1906, vol. 76, pp. 493, 494.)—Results of analyses are given showing that the camphor is completely volatilized from camphor liniment by heating a thin layer of the latter in a shallow, flat-bottomed basin for one hour on the water-bath. The size of the basin employed should be such that about 4 grams of the oil form a layer in it 1 mm. in depth. Under these conditions the olive oil itself does not gain in weight. W. P. S.

On a Reaction of Morphine. C. Reichard. (*Pharm. Centralh.*, 1906, xlvii., 247; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 142.)—The formaldehyde reaction for morphine is usually carried out in the presence of concentrated sulphuric acid; the author finds it better to add a trace of stannous chloride instead of the acid. As modified thus, the reaction is a delicate test for either morphine or tin. A. G. L.

A New Reaction of Morphine. D. Radulescu. (*Bull. Soc. Sci. d. Bucurcsci*, 1905, 14, 602; through *Pharm. Journ.*, 1906, vol. 76, p. 501.)—The following colour reaction is stated to be peculiar to morphine: A filtered decoction of the substance to be examined, as free as possible from colour, is treated with a crystal of sodium nitrite and sufficient acid to liberate the nitrous acid. Before effervescence has ceased an excess of potassium hydroxide solution is added. In the presence of morphine a pale rose to ruby-red tint develops, according to the amount of morphine present. The kind of acid employed is immaterial, and any alkali may be used. The red colour is not removed by shaking with immiscible solvents nor discharged by boiling the alkaline solution, but is decomposed by strong acids. It also decomposes in the presence of much alcohol. W. P. S.

On the Precipitation and Quantitative Determination of Alkaloids by means of Solutions of Potassium Bismuth Iodide. D. Jonesen. (*D. pharm. Ges. Ber.*, 1906, 130; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 156.)—Thoms has shown that potassium bismuth iodide may be used, not only to detect organic bases, but also for the precipitation of certain alkaloids. The reagent is added to a sulphuric acid solution of the alkaloid, and the precipitate washed with dilute sulphuric acid. From the precipitate the alkaloid may be recovered by treatment with a mixture of sodium carbonate and hydrate (which treatment requires to be prolonged in some cases). In this way, from 1 gram each of quinine, caffeine, and antipyrin, 0·9405, 0·9546, and 0·9273 gram respectively was recovered, showing the precipitation with the potassium bismuth iodide reagent to be fairly complete.

A. G. L.

ORGANIC ANALYSIS.

The Determination of Methyl Alcohol in Formalin. O. Blank and H. Finkenbeiner. (*Berichte*, 1906, xxxix., 1326, 1327.)—Methyl alcohol is an ordinary constituent of the commercial (40 per cent.) solutions of formaldehyde, an addition of 12 to 18 per cent. being made to prevent the liquid becoming permanently turbid when exposed to cold. The author bases a method for its determination on the amount of oxygen consumed when a weighed quantity of the formalin is oxidized by means of chromic acid and sulphuric acid—

$$H.COH + O_2 = CO_2 + H_2O$$
$$CH_3.OH + 3O = CO_2 + 2H_2O.$$

One gram of the sample is introduced into a mixture of 50 c.c. of $\frac{N}{2}$ chromic acid solution with 20 c.c. of strong sulphuric acid, and the whole allowed to stand for about twelve hours at the ordinary temperature, or about one to one and a half hours if the liquid be heated cautiously, care being taken not to let it evaporate below a third of its volume.

The liquid is then made up to a litre and 50 c.c. withdrawn, and, after the addition of a crystal of potassium iodide, titrated with a $\frac{N}{10}$ sodium thiosulphate solution. From the result the total amount of oxygen consumed can be calculated, and, the amount of formaldehyde in the original solution being known, the percentage of methyl alcohol can be obtained indirectly. In a test experiment described the result obtained was identical with the amount (7·65 per cent.) of methyl alcohol added to a 20 per cent. solution of formaldehyde. C. A. M.

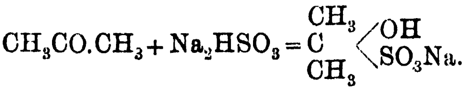
New Method for the Determination of Acetone. S. J. M. Auld. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 100, 101.)—The acetone in wood spirit, crude acetone, etc., is converted into bromoform, the latter decomposed with alcoholic potash, and the residual potassium bromide titrated with silver nitrate solution, the hydrolysis being represented by the equation—

$$3CHBr_3 + 9KOH + C_2H_5OH = 3CO + C_2H_4 + 9KBr + 7H_2O.$$

A quantity of the solution containing 0·1 to 0·2 gram of acetone is pipetted into a 500 c.c. round-bottom flask, diluted with a little water, and mixed with 20 to 30 c.c. of a 10 per cent. potash solution. The flask is attached to a long reflux condenser, and is fitted with a dropping funnel containing a solution of 200 grams of Br and 250 grams of KBr per litre. This solution is allowed to flow into the mixture until it has acquired a faint yellow tinge, the flask heated on the water-bath at about 70° C. for half an hour, and more bromine solution added drop by drop until the slight coloration is permanent, any excess being removed by boiling with a little more potash. The mixture is then distilled until the distillate is free from bromoform, water being added to the contents of the flask if necessary, and the condenser rinsed with a little alcohol. Distillate and washings are mixed with 50 c.c. of alcohol and sufficient solid caustic potash to make an approximately 10 per cent. solution, and the mixture heated on the water-bath under a reflux condenser until the bromoform is completely decomposed, which occupies about three-quarters of an hour. After evaporating to smaller bulk, if necessary, the liquid is cooled, neutralized with dilute nitric acid, diluted to 500 c.c., and an aliquot part titrated with $\frac{N}{10}$ silver nitrate, using K_2CrO_4 as indicator; 240 parts of bromine correspond to 58 parts of acetone. Pure bromine must be employed, as crude bromine frequently contains bromoform.

W. H. S.

The Determination of Acetone. A. Jolles. (*Berichte*, 1906, xxxix., 1306, 1307.)—The method is based upon the fact that acetone combines with sodium bisulphite (1 molecule : 1 molecule), forming the sodium salt of a sulphonic acid similar to that obtained with furfural (ANALYST, xxxi., 116)—

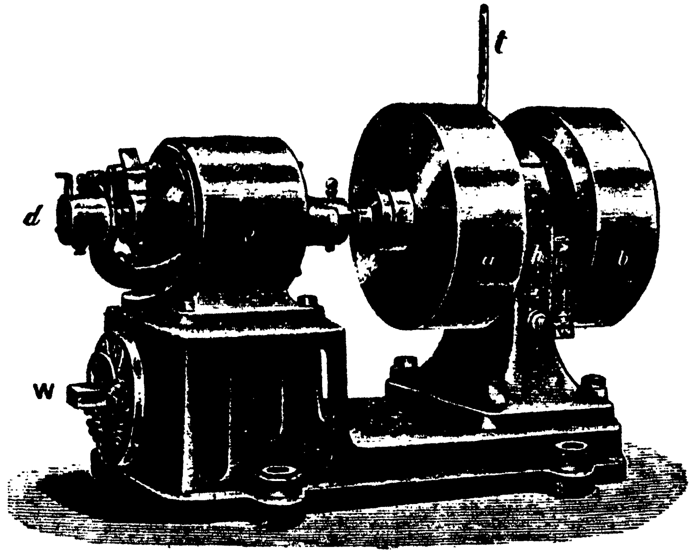


$$CH_3CO.CH_3 + Na_2HSO_3 = \overset{CH_3}{\underset{CH_3}{C}} \begin{matrix} \diagup OH \\ \diagdown SO_3Na. \end{matrix}$$

The standard bisulphite solution is added to the acetone solution in a quantity three or four times greater than the theoretical amount, and the excess is titrated with standard iodine solution after the mixture has been allowed to stand for not less than thirty hours. A blank determination with the sodium bisulphite solution should be made simultaneously to obtain the correction for any changes it may undergo during the experiment.

C. A. M.

Experiments made with Dettmarsch's Apparatus for Testing Oils. Richard Kissling. (*Chem. Zeit.*, 1906, xxx., 152.)—The author has made over 500 tests on the Dettmarsch apparatus (shown in the figure) for testing the lubricating power of oils. The apparatus consists essentially in two heavy fly-wheels, *a* and *b*, connected by a shaft revolving in a journal between them, to which the oil to be tested is fed from the reservoir (*h*, situated just under the right-hand bearing), where it is heated electrically. The fly-wheels are driven by the motor, *c*, and can be instantaneously disconnected from the latter by releasing a spring, *d*. The method of working is to impart a certain measured velocity to the fly-wheels, then to release the spring, and to measure the time taken for the revolution to stop entirely. From these data a "coefficient of friction" is calculated. Although the apparatus often gives results in good agreement with the viscosity determinations and the results obtained in practice, the author has obtained so many totally inexplicable results with it (apparently attributed by him to the presence of an imperfection in the apparatus) that he condemns it entirely. He believes the only way to determine the lubricating power of an oil to be to test it *in situ* in the machine for which it is to be used.



A. G. L.

Preparation of Aldehyde-free Ethyl Alcohol for Oil and Fat Analyses. F. L. Dunlap. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 395-398.)—Alcohol yielding a colourless potash solution may be obtained by adding to 1 litre in a glass-stoppered cylinder a solution of 1·5 grams of silver nitrate in 3 c.c. of water, mixing thoroughly, then pouring in slowly, and without shaking, a solution of 3 grams of potash in 10 to 15 c.c. alcohol. The finely-divided silver oxide precipitated gradually distributes itself through the alcohol, and after standing overnight completely settles, when the clear, supernatant liquid is filtered or siphoned off and distilled.

W. H. S.

On the "Milkiness" of Paraffin Wax. H. Mittler and R. Lichtenstern. (*Chem. Rev. Fett. u. Harz-Ind.*, 1906, xiii., 104, 105.)—Transparent paraffin wax fetches a higher price than "milky" wax, and the greater value of the clear products is due to more than their better appearance. Neustadtl concluded that "milkiness" was caused by differences in the melting-points of the different constituents of the paraffin ; but the authors contest this, and point out that transparent paraffins with melting-points as far apart as 57° and 42° C. can be mixed together without rendering the product opaque, though in some instances a bluish shade may be produced. They attribute the "milkiness" to the presence of paraffin oil, and have isolated that oil from "milky" paraffin by extraction with a mixture of alcohol and ether, and have rendered transparent paraffins "spotted" or "milky" by adding oil in the proportion of 0·5 to 3 per cent. The addition of smaller quantities up to 0·3 per cent. has little or no effect upon the transparency. C. A. M.

Constants of Carnaüba Wax. L. G. Radcliffe. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 158.)—The following figures have been obtained with a sample of Ceará wax: Melting-point (in capillary tube), 84° C.; acid value, 2·9; acid value determined by dissolving 3 grams in 120 c.c. of boiling amyl alcohol, 5; saponification value (5 grams dissolved in 60 c.c. amyl alcohol, and boiled for six hours with 50 c.c. alcoholic potash containing 60 grams of potash per litre), 88·3; ester value, 85·4; iodine value, 13·17. The bleached wax melted at 61°, and had an acid value in amyl alcohol solution of 0·56. W. H. S.

On the Proportions of Pentosan and Methylpentosan in Plant Products. John Sebelien. (*Chem. Zeit.*, 1906, xxx., 401.)—P. Sollied has continued the work done by the author and others to show that in the great majority of cases the amount of methylpentosan occurring in plants is considerably less than that of the pentosan also present. Sollied's results are given in the following table, Tollens and Ellett's method (*Ber.*, 1905, 492) having been used:

	Methylpentosan, Per Cent.	Pentosan, Per Cent.
Oak-wood, fifteen-year-old tree	2·26	19·06
Ditto, younger	2·31	18·60
Oak-bark, eighteen-year-old tree	2·08	14·21
Ditto, younger branches	2·54	12·85
Cedar-wood	2·90	12·36
Fir-wood	4·70	10·03
Beech-wood	2·68	23·59
Ash-wood	2·95	17·24
Hay	2·13	17·43
Fucus vesiculosus	3·16	6·32
Ascophyllum nodosum	3·47	8·46
Rye-bran	1·75	20·93
Oats	1·09	12·76
Rape-seed cake	1·72	6·25
Linseed cake	2·62	9·73
Cotton-seed cake.	1·72	6·23
Carrots (dry substance)	2·59	8·43
Kohlrübe (ditto)	2·93	6·67

A. G. L.

Linaloe Oil. W. Naumann. (*Chemist and Druggist*, 1906, lxviii., 619.)—The following results were obtained on the analysis of three samples of oil shipped direct from Mexico and guaranteed by the shippers to be pure oils :

	No. 1.	No. 2.	No. 3.
Specific gravity at 15° C.	0·8785	0·8800	0·8797
Rotation in 100 mm. tube	- 8° 30′	- 12°	- 11° 10′
Refractive index at 20° C.	1·4635	1·4633	1·4618
Esters	4·3%	4·7%	4·9%
Linalol	66%	68%	63%
Distilling below 190° C.	none	none	
,, ,, 195° C.	7%	5%	—
,, ,, 200° C.	25%	35%	—
,, ,, 205° C.	45%	40%	—

These results are similar to those obtained by Parry and Bennett (ANALYST, 1906, 201), and accordingly the above oils belong to the best class now obtainable.

W. P. S.

On the Chemical Examination of Kerosine. K. Charitschkow. (*Westnik shirow.*, vii., 14 ; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 142.)—Dehnstedt and Geisler have recently shown the presence of neutral sulphuric esters in various petroleum oils imported at Hamburg. The author now shows that similar esters are sometimes present in Russian petroleum, the quantity varying inversely with the amount of acid used in the purification of the oil. The esters may be recognised by treating the oil with aniline, aniline sulphate being insoluble in kerosine. On burning oil containing sulphuric esters, sulphurous and sulphuric acids are formed and the lamp-wick is charred.

A simple method of determining sulphur in petroleum consists in distilling it over potassium, potassium sulphide being formed and determined in the usual manner. The quantity of unsaturated hydrocarbons present in kerosine should always be determined, as they yield a flame of unpleasant colour. A. G. L.

Collaborative Work on Tannin Analysis. F. H. Small. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 296-298.)—The American Leather Chemists' Association have formulated the following methods for the estimation of tannin in raw materials and analysis of the tannin solutions obtained therefrom. The material is ground to a specified fineness, and the tannin extracted in a copper Soxhlet apparatus so constructed that the extractive is removed from the action of heat, as prolonged boiling of the liquor converts part of the tannin into some compound not tannin, probably gallic acid. The analysis of the tannin solution is conducted on a solution containing 0·35 to 0·45 gram of tannin per 100 c.c. The residues obtained from the evaporation of 100 c.c. each of the solution before and after filtration give the amounts of total and of soluble solids respectively. The tannin is removed from another portion of the solution by treatment with hide-powder, which is then filtered off, and the clear filtrate evaporated to dryness, the residue being the soluble non-tannins. By deducting these from the total soluble matter the proportion of tannin is readily obtained. The hide-powder employed is first treated with a dilute solution of chrome alum, washed free from uncombined salts, excess of water squeezed out, and the moist powder added to the tannin solution, a correction being made for the water added with the hide-powder. Difficulties have arisen owing to failure to obtain clear filtrates, and to the retention of part of the tannin by the filter-paper, but by following prescribed conditions concordant results are obtainable. To avoid decomposition during evaporation and drying of the residues, a special copper steam-bath has been devised, with a solid top on which the dishes rest, and fitted with a steam-jacketed cover provided with openings for ventilation. W. H. S.

Analysis of Ebonite Articles. R. Thal. (*Chem. Zeit.*, 1906, xxx., 499.)—As supplied to the Russian army medical service, ebonite cases for instruments and bottles must fulfil the following requirements: The ebonite must be made from best Para rubber and sulphur. The quantity of the latter may vary from 27 to 30 per cent., but should preferably be 29 per cent. The vulcanization must be even throughout, and the ebonite must be free from surrogates. The amount of ash must not exceed 0·60 per cent. On warming the ebonite with fuming nitric acid, a clear or nearly clear solution should be obtained. The material should be black, capable of being polished, and be free from cracks, indentations, porous or coloured spots, etc.; it must be sufficiently hard, and yet not too brittle to be worked by tools. The cases must not break or crack on allowing them to fall by their own weight when empty on to a stone floor, or on knocking them together.

The analysis of ebonite is carried out by the methods usual for rubber. Moisture may be determined by drying for two hours at 100° to 105° C.; longer drying results in oxidation of the material. For the test with fuming nitric acid, 0·5 gram of the ebonite in small pieces is gradually added to 20 c.c. of the acid, and the whole is warmed until nitrous fumes are driven off. A turbid solution indicates an amount of ash greater than 0·6 per cent. In testing for surrogate, a correction must be made for the weight lost by the filter-paper on treatment with 8 per cent. alcoholic caustic soda. The author has previously found this to be 5·5 per cent. For accurate work corrections must also be made for the amount of sulphur extracted from, and the quantity of soda retained by, the dried residue. A. G. L.

Grading of Glues and Gelatines. J. Alexander. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 158-161.)—The author recommends the determination of the reaction to litmus, viscosity, grease, foam, "set," jelly strength, adhesive strength (under actual working conditions), and keeping properties. The viscosity is measured by noting the time of efflux at 80° C. from a 45 c.c. pipette of solutions containing 25 grams of glue in 100 c.c. of water. The effluent tube is 7 cm. long, and has external and internal diameters of 9 and 6 mm. respectively, the smallest diameter of the outlet being about 1·5 mm. The length of the bulb is 9·5 cm., and its outside diameter 3 cm. The amount of grease is approximately proportional to the spots appearing when the glue solution is painted on white paper with a little aniline or dry colour.

Foam is estimated comparatively by agitating with a rod or mechanical agitator, and "set" is the speed with which the jelly hardens. Jelly strength is well measured by pressure with the finger-tips; but a special instrument, more reliable than the shot test, has been devised for the purpose, consisting of a brass cylindrical vessel supported like a gas-tank by four vertical rods, against which it slides with almost frictionless roller-bearings. This rests on a truncated cone of jelly, and shot are poured in until a definite expression of jelly is observed, two vertical brass uprights 3·5 cm. high connected with an electric-bell circuit being placed beneath the cup, so that when this reaches their level the bell rings. Keeping properties are observed by exposing the uncovered glue solutions for some days at room temperature. An arbitrary series of standards has been devised, based upon the viscosities, sixteen nearly equidistant grades having values assigned of 10 to 160, 10 points being allowed between each grade. The grades specially adapted for particular purposes are mentioned. W. H. S.

Conditions affecting the Foaming and Consistency of Glues. S. R. Trotman and J. E. Hackford. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 104-107.)—Foaming of glue is increased by overboiling, by prolonged boiling with alkalies, and by the presence of peptones, of soda or lime, of soap beyond a certain limit, of carbolic acid, and of suspended matter. From the influence of the last-named, loaded glues, such as Russian, may be expected to give considerable foam. The amount of foam is reduced by rise of temperature, by the addition of lubricating, bone, or cod oil, or of hydrochloric, sulphuric, acetic, salicylic, or oleic acid. The mineral constituents of water used in glue-making would not affect the quality of the resulting glue. The consistency of glue is not injured by small quantities of peptone so long as they do not replace albumose nitrogen, but is seriously reduced by alkalies and acids, though in a less degree by small quantities of carbolic or boric acid. A small amount of soda or potash soap does not diminish the consistency. The foaming has been measured by means of a graduated tube about 70 cm. long, and of such diameter that each division is 1 cm. in length and has a capacity of 1 c.c. This is half filled with a 10 per cent. solution of the glue, placed in a water-jacket at 60° C., and after allowing the glue solution to attain this temperature, withdrawn, and the level of the solution adjusted to zero. The tube is then corked and shaken vigorously for one minute, replaced in the water-jacket, and the height of the foam read off. Consistency has been determined by noting the time taken by spheres to fall through a column of the given glue solution. H. J. Watson (*ibid.*, 1906, xxv., 209-211), from independent experiments on the same lines, concludes that foaming is not materially increased by the presence of free alkali or acid, except sulphurous acid. Mucins increase the foam, and zinc sulphate lengthens the time it takes to die away, but heat appears to be the all-important factor in producing foaming glue, probably due to hydrolysis of the gelatin to gelatone. By reducing the period under heat to the shortest possible, practically foam-free glue should be obtained. This author measures the foam by shaking 50 c.c. of a 10 per cent. solution of the glue at about 50° C. in a stoppered graduated cylinder 2·5 cm. in diameter, and reading the height of the top of the foam as soon as the height of the solution reaches 45 c.c. W. H. S.

The Determination of Urea. B. Glassmann. (*Berichte*, 1906, xxxix., 705-710.—The following modification of Liebig's and Pflüger's methods is claimed to give very accurate results, and to be more easily applied than any other method. The urea is precipitated from the solution, which has been neutralized with sodium carbonate, by means of a known excess of standard solution of mercuric nitrate, the precipitate filtered off, the filtrate acidified with nitric acid, and the excess of mercury determined by titration with $\frac{N}{10}$ ammonium thiocyanate solution, iron ammonium sulphate being used as indicator. From the amount of mercury that has been taken up the urea is calculated by means of the equation—

$$2CON_2H_4 + 4Hg(NO_3)_2 + 4H_2O = [2CO(NH_2)_2 + Hg(NO_3)_2 + 3HgO] + 6HNO_3.$$

The conditions for the determination of the urea in urine by this method are the same as those required in Liebig's original method—viz.: (1) The urine must not contain more than traces of albumin, which otherwise must be precipitated with acetic acid. (2) It must not be in full alkaline fermentation. (3) It must not contain leucine or tyrosine. (4) It must not contain abnormal amounts of alkali chlorides, bromides, or iodides, or of benzoic, salicylic, or hippuric acids. (5) If too darkly coloured it should be treated with animal charcoal. C. A. M.

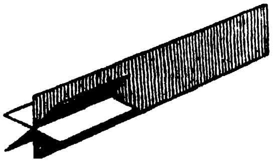


FIG. 1.

Simplified Ultimate Analysis for Technical Purposes—II. M. Dennstedt. (*Zeits. angew. Chem.*, 1906, xix., 517; *cf.* ANALYST, xxx., 135, 348, and this vol., 122.)—The author has given up the use of platinized quartz, which occasionally scratches the combustion-tube, and now uses rolled-up pieces of platinum foil, or, better, platinum-foil "stars" (Fig. 1), weighing 1 or 2 grams, which may be obtained from Hereaus, of Hanau. For the introduction of the double current of oxygen, the arrangement shown in Fig. 2 is recommended, as it very seldom requires renewal. A. G. L.

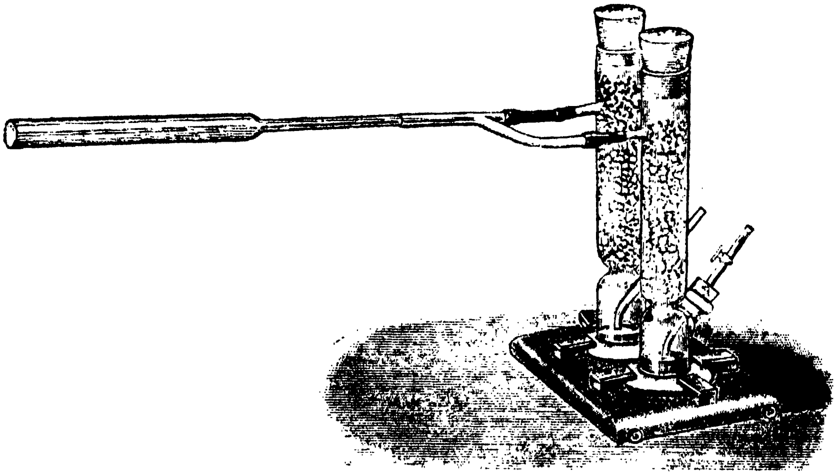


FIG. 2.

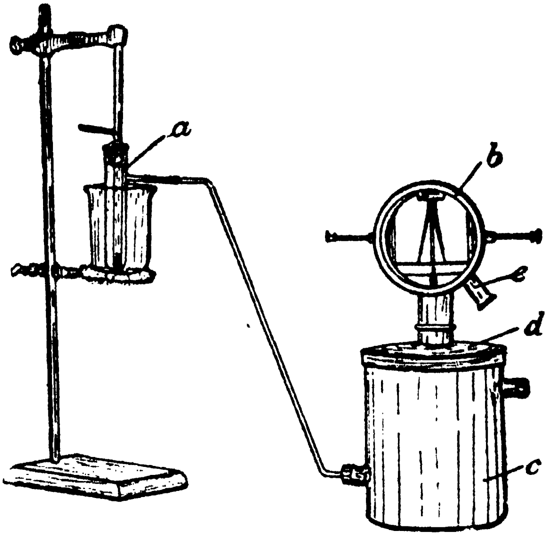
INORGANIC ANALYSIS.

On the Quantitative Determination of Arsenic by the Marsh Method. G. Bertrand and Z. Vamossy.

(*Ann. Chim. Phys.*, 1906, 523; through *Chem. Zeit.*, 1906, xxx. [*Rep.*], 141.)—The method gives good results, provided that the authors' instructions (*Ann. Chim. Phys.*, 1903, 242) are used with the modification that a length of 40 cm. of the tube is heated and that the tube has a bore of 2 mm. For quantities of 0·01 gram arsenic and more, the gas, after leaving the apparatus, is passed through a solution of silver nitrate. The arsenic mirror should be dissolved in sodium hypochlorite and not in nitric acid. A. G. L.

On a Method for the Detection of Very Small Quantities of Yellow Phosphorus. R. Schenck and E. Scharff.

(*Ber.*, 1906, 1522; through *Chem. Zeit.*, 1906, xxx., [*Rep.*], 156.)—Yellow phosphorus, even in very small quantities, ozonizes air, and hence may be made to discharge an electroscope; phosphorus trisulphide, even when luminescent, does not possess this property, and does not yield any phosphorus trioxide on slow combustion at moderate temperatures. The authors make use of the apparatus shown for the detection of small amounts of yellow phosphorus. By means of a tube passing through the ground-glass stopper of the tube (*a*) a slow current of air is led over the substance to be examined, which is placed at the bottom of the tube. The air then passes through the vessel (*c*), which communicates by means of its removable stopper (*d*) with an electroscope (*b*). A piece of sodium in the tube (*e*) serves to keep the electroscope dry and sensitive. A. G. L.

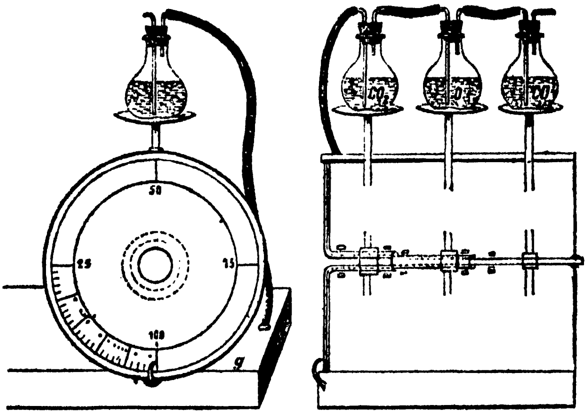


Determination of Small Quantities of Copper in Water. E. B. Phelps.

(*Journ. Amer. Chem. Soc.*, 1906, xxviii., 368-372.)—The copper is separated electrolytically from an acid solution, dissolved in dilute acid, and, after neutralizing, determined colorimetrically as sulphide. Sufficient water to contain 0·1 to 1·0 mgm. of copper is evaporated to 75 c.c. and washed into a platinum dish, which serves as anode, adding 2 c.c. of dilute sulphuric acid for clear and soft waters, 5 c.c. for those rich in organic matter or clay. The cathode, consisting of a flat spiral of stout platinum wire, is then suspended in the solution parallel to and about $\frac{1}{2}$ inch from the bottom of the dish, and the circuit closed. After electrolysing for four hours with occasional stirring, using a current of 0·02 ampère, the cathode is removed without first opening the circuit and immersed in dilute nitric acid previously heated to boiling. The spiral is washed and the liquid evaporated to dryness on the water-bath, a few drops of hydrochloric acid being added if silver is suspected. The residue is then dissolved in water, washed into a Nessler glass, made up to 100 c.c., and 10 c.c. of an alkaline solution of potassium sulphide, prepared by mixing equal volumes of 10 per cent. potash solution and a saturated aqueous solution of sulphuretted hydrogen, added. A similar tube is prepared by adding 10 c.c. of the reagent to a tube of distilled water, and then running in, 0·2 c.c. at a time, a standard solution containing 0·2 gram copper per litre. This method gives good results in the presence of sewage and industrial waste, iron, lead, silver, and tin, and in highly-coloured surface-waters. W. H. S.

APPARATUS.

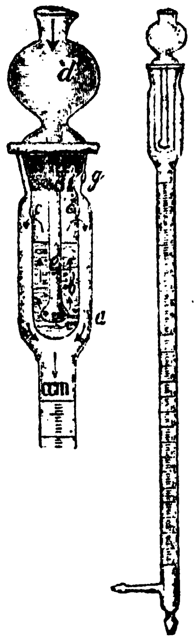
Apparatus for the Continuous Registration of the Results of Gas Analysis and Precipitation in Liquids. (*Chem. Zeit.*, 1906, xxx., 347.)—B. Stollberg has patented (in Germany) the apparatus shown in the figure. It consists essentially of a number of flasks, containing suitable reagents, connected with each other and mounted on separate pendulum - balances, which rotate graduated paper discs at speeds corresponding with their weights. On these discs a pencil worked by clock-work marks off minutes. By means of a pump (not shown) worked by a falling-weight mechanism determined quantities of the gas (from a chimney, etc.) to be analysed are drawn through the flasks, and the increase in weight of each flask in a given time is read off from the discs. By means of suitable modifications the apparatus may also be made to give a continuous record of the specific gravity of a liquid, the amount of suspended or precipitated matter in it, and the like. A. G. L.



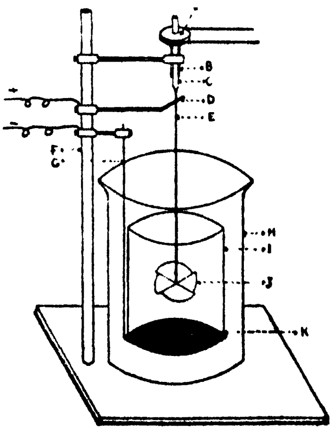
Modified Westphal Balance for Solids and Liquids. **F. M. Williams.** (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 185-187.)—The balance is adapted for solids, particularly cements and minerals, by lengthening the pointer, upon which, at a distance equal to the length of the other arm, is suspended a support carrying two pans, one above, the other below, the surface of the liquid, by means of which the object may be weighed first in air, then in the liquid. A two-piece counterpoising weight movable along the threaded portion of the pointer facilitates adjustment, and a cam device for raising the beam and locking it in place tends to prevent wear upon the knife edges. The insertion of a hinge-joint in the upright standard arm allows the instrument to be folded up within a small space. W. H. S.

A Burette Top for the Absorption of Carbon Dioxide and other Gases (*Chem. Ztg.*, 1906, xxx., 459).—Gustav Müller, of Ilmenau, has devised the apparatus shown, which is designed to prevent the absorption of carbon dioxide by caustic alkali solutions in the burette. The apparatus fits in the top of the burette, which is widened to admit it, by means of a ground-glass joint. It contains a solution of caustic alkali, through which any air entering the burette has to bubble, the path of the air being shown by the arrows. A piece of solid potash may also be placed in the bulb (*d*). In filling a burette for the first time with the standard solution, the apparatus is placed on the top of the burette and filled with potash. It is then turned so that a groove (*f*) at the ground-glass joint is opposite a hole (*g*) in the burette. The standard solution is then run in from below, the air from the burette escaping through (*g*). The top is then turned so as to shut off communication between (*g*) and (*f*), and kept in this position. Any air entering the burette has then to bubble through the potash, whilst in filling the burette afresh the potash is simply forced up into the bulb (*d*).

A. G. L.

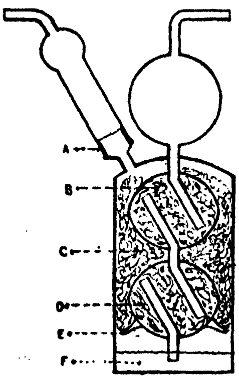


A New Electrolytic Apparatus. S. F. Acree. (*Amer. Chem. Journ.*, 1906, vol. 35, pp. 313-316.)—The essential feature of the apparatus is that by means of a rotating, propeller-like anode all the liquid is continually circulated through the cathode K, which is of platinum gauze, 50 meshes to the linear inch, and consists of an outer ring 2·5 inches in diameter, forming one piece with the arm G, and an inner removable ring which holds the gauze in position, the two rings being kept firmly together either by fine platinum wire or by four small drops of gold. The arm G is held in position by a binding-screw attached to the glass rod F, and supports K half an inch above the bottom of the beaker H. A glass cylinder, I, rests on the outer ring of K, its upper edge being at least a quarter of an inch beneath the surface of the liquid. The anode J, of heavy platinum foil, is riveted to the platinum rod E, which is sealed into the glass tube C, and the latter rotated in the glass tube B by means of a pulley, A, and a Rabe water turbine or a small electric motor. A platinum strip or stout wire, D, connected to a binding-screw on F brushes against E, making good contact. The distance between J and K is adjustable, and depends entirely upon the voltage and strength of current required. W. H. S.



New Form of Potash Bulbs for Estimation of Carbon Dioxide from Combustions. S. F. Acree. (*Amer. Chem. Journ.*, 1906, vol. 35, pp. 309-311.)—

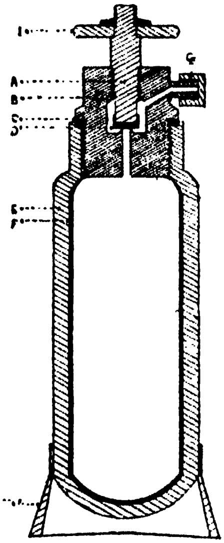
A modification of Soxhlet's alkali apparatus, in which the two bulbs B and D are filled and surrounded with glass wool saturated with 50 per cent. KOH solution, the mixture of gases from the combustion-tube being thereby brought in contact with a very large surface of alkali solution, insuring almost perfect absorption. The glass wool around the bulbs is kept in position by four small glass projections E, which extend from the lower bulb nearly to the wall of the outer vessel, and the exit tube from the lower bulb dips into the potash solution F. A calcium chloride tube is attached by means of a ground-glass joint at A. The efficiency of the apparatus is proved by the fact that on passing through two of them in series (*a*) a stream of nearly pure CO_2 for fifteen minutes, (*b*) a mixture of one-third CO_2 and two-thirds air, in neither case was the amount of CO_2 absorbed by the second apparatus as much as 0·10 mgm. W. H. S.

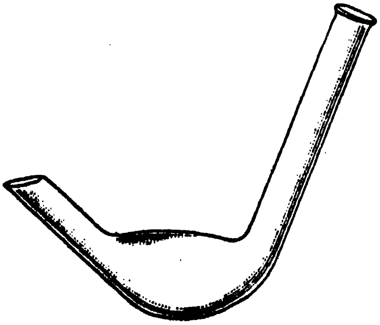


Porcelain-lined Bomb for General Laboratory Use. S. F. Acree. (*Amer. Chem. Journ.*, 1906, vol. 35, p. 309.)—

The bomb E is of soft steel, about $\frac{3}{8}$ inch thick, and tested to 250 atmospheres water pressure, its entire inner surface up to the threads at the neck being lined with white porcelain $\frac{1}{16}$ inch thick, which may be of acid or of alkali-proof material. The stopper B is of hard bronze, has a square head, and screws into the neck of the bomb with heavy threads, the ring C, of very hard rubber or lead, making a gas-tight joint. The handle I, attached to the screw A on a square head, serves as a valve, the screw fitting into the stopper with gas-tight threads and packing, and having a tip, D, of very hard rubber or lead, which closes the bomb so tightly that liquid carbon dioxide cannot escape. The inlet or outlet G has a cap, and is threaded to fit carbon dioxide cylinders. The bomb rests in a detachable ring base, H.

W. H. S.



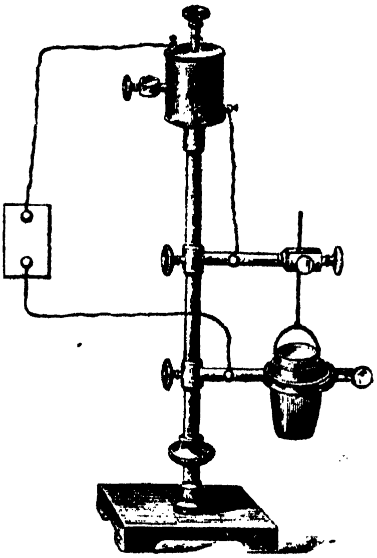


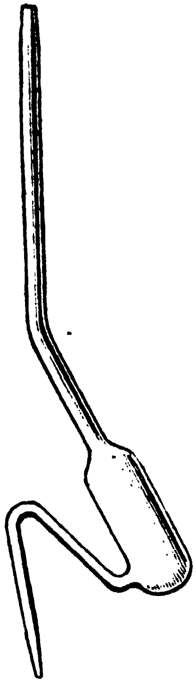
Dougal Assay Tube.—This, which is a modified flask, will be found useful for "parting," dissolving metals, minerals, etc., solution proceeding quietly, as the double opening provides a free escape for the evolved gases. The apparatus may be obtained from Messrs. J. J. Griffin and Sons.

W. J. S.

A New Rheostat for Electrolytic Analysis.

G. Pascalis. (*Mon. Scient.*, 1906, 4 Ser., 20, 168; through *Chem. Zeit*, 1906, xxx. [*Rep.*], 104.)—The small adjustable resistance shown in the figure on the top of the electrolytic stand has been designed by the author in conjunction with Grésy. Its height is only 80 to 100 mm., the diameter being 50 to 60 mm. A casing protects it from fumes. By means of the screw on the top the strength of a current at 4 to 6 volts may be regulated from 0 to 1 ampère in $\frac{1}{10}$ ampère stages. A. G. L.





Struthers Syphon Pipette possesses the following advantages—compactness, less danger of liquid pipette being sucked up into the mouth, no necessity for using tip of finger in stopping flow, mark more exactly and easily caught, easy regulation and stoppage of flow, which is effected by simply tilting the pipette; it always discharges the same volume, as the bulb completely empties itself. The pipette is manufactured by Messrs. J. J. Griffin and Sons.

W. J. S.

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REVIEWS.

The Cyanide Industry. By R. Robine and M. Lenglen. Translated by J. Arthur Leclerc, Ph.D. With an Appendix by C. E. Munroe, Ph.D. (New York: Wiley and Sons. London: Chapman and Hall, 1906. Price 17s. net.)

The manufacture and utilization of cyanogen compounds have undergone rapid development in recent years, and the work under review is an attempt to classify the various methods employed at the present time. The book comprises four subdivisions, the first of these dealing with the chemical reactions involved, together with the preparation and properties of the more important single and double cyanides. Part II. is practically a directory of the works at which the cyanide industry is carried on, whilst Part III. is devoted to the manufacturing processes in general use.

The commercial application of the different cyanogen compounds is discussed in the last section, and an appendix contains a digest of the United States patents relating to the recovery of precious metals by means of cyanides.

The descriptive matter as a rule is concise, and the processes of manufacture are clearly explained. The authors point out that the application of cyanides to the treatment of auriferous materials is the immediate cause of the progress realized by the industry during the last fifteen years, whilst the discovery of cyanide compounds in the bye-products of the manufacture of illuminating gas has also widened the field of investigation towards synthetic processes.

In the small space allotted in the book to analytical properties the usual tests for cyanides are included, but the information given in reference to quantitative determinations is rather meagre.

Although the book will probably prove useful to the manufacturer as a résumé of the ordinary methods employed in the production of cyanides, its value to the analytical chemist is somewhat problematical. P. A. E. R.

Les Industries de la Conservation des Aliments (The Food-preserving Industry). By X. Rocques. (Paris, 1906. Pp. xii + 506. Price 15 francs.)

This book is divided into seven chapters, respectively treating of historical sketch, the causes and effects of changes due to putrefaction, preservation by heat, by cold, by desiccation, by antiseptics, and by the prevention of the entrance of micro-organisms into such naturally sterile products as eggs.

There is little or no analytical information, the whole subject being treated of from a technical point of view; but the work is well up to date, and gives a fairly complete description of the various methods of food preservation. As is natural in a French work, the processes and details of packing and transport are chiefly those used in France, but these are fairly typical, and in most cases will give the food analyst a good general idea of the technology of the food-preserving industry.

One passage taken from the report of Dr. Bordas, adopted by the Congress of Hygiene, is worth quoting *in extenso :*

"En résumé, l'addition d'antiseptiques dans les denrées alimentaires produit les resultats suivants:

"1. Elle est susceptible de nuire à la sante.

"2. Elle peut permettre de conserver des éléments ayant déjà subi un commencement d'altération.

"3. Elle modifie le plus souvent la composition des éléments organiques.

"*Il y a lieu d'interdire l'emploi des antiseptiques quels qu'ils soient, nocifs ou non, dans toutes les matières alimentaires.*" H. D. R.

AUGUST, 1906. Vol. XXXI., No. 365.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON THE PRESENCE AND DETECTION OF CYANOGEN IN JAVA, BURMA, AND HARICOT BEANS.

BY R. R. TATLOCK AND R. T. THOMSON.

(*Read at the Meeting, May* 2, 1906.)

As we had to deal with this question in the recent poisoning of cattle by "Java" beans, we have thought it might be of interest to put on record the result of our examination for cyanogen compounds of "Java" and other beans, as well as of peas of various kinds. As a fairly full report with regard to the poisoning cases in Scotland is given in the March and April numbers of the *Journal of the Board of Agriculture*, we shall not refer further to these, nor, indeed, to anything directly connected with them, but shall confine our attention to the various specimens which we procured for experiment.

For the determination of cyanogen we employed the method which Professor Dunstan used — namely, extracting the glucoside with alcohol, distilling off the latter, decomposing the former with hydrochloric acid, and distilling off the hydrocyanic acid (the *Agricultural Ledger*, No. 2, 1905). For the detection of the hydrocyanic acid we made a mash of about 10 grams of the ground beans with 20 or 30 c.c. of warm water in a small stoppered bottle and kept at 40° to 50° C. for half an hour, then applied a modification of the Schönbein test. This consisted in holding in the vapour in the bottle a piece of filter-paper, which had been first dipped in a 0·2 per cent. tincture of guaiacum resin, and then in a 0·1 per cent. solution of copper sulphate, when the paper assumed a fine blue colour almost immediately, if hydrocyanic acid was present. It is important that the blue colour should be rapidly developed, as the prepared paper slowly assumes a blue tint when exposed to the air, and this might be mistaken by careless observers for the effect of hydrocyanic acid. A good plan, therefore, is to prepare two slips of paper simultaneously, insert one into the vapour to be tested for hydrocyanic acid, and simply expose the other to the air, when a comparison will decide the question. If this plan is adopted no mistake can be made. In applying the Schönbein test it is generally directed to dip the filter-paper in a 10 per cent. tincture of guaiacum resin, and dry before dipping in the 0·1 per cent. solution of copper sulphate. On drying the paper, however, it always assumes a blue tint, and this vitiates its application to the detection of hydrocyanic acid. In consequence of this defect we have modified the test as described above.

It should be noted that, as a rule, hydrocyanic acid (if present) can be detected after half an hour's warning; but where no positive result is obtained, the bottle should be closed and allowed to stand at the ordinary temperature for several hours, in order to allow the hydrocyanic acid to accumulate by the gradual action of the enzyme contained in the bean on the cyanogenetic glucoside.

After deciding the method of procedure, we, in the first place, examined several specimens of Java beans, and found from 0·027 to 0·137 per cent. of hydrocyanic acid, this being, of course, understood to exist in the beans in the form of an amygdalin-like glucoside. These results practically agree with those of the uncultivated variety examined in the Scientific and Technical Department of the Imperial Institute. In order to determine whether there was really any difference as regards cyanogen contents in beans of different colour, we obtained an average sample, weighing about 3 pounds, of a lot of Java beans, and separated them into five different kinds. These consisted of white, brown of various shades, speckled, chiefly brown and white, with a few purple and white, purple of various shades, and black beans. The sample was composed of these as follows:

	Per Cent.
White beans	9
Brown beans	34
Speckled beans	33
Purple beans	20
Black beans	4
	100

These were tested separately with the following results:

	Hydrocyanic Acid. Per Cent.
White beans	0·027
Brown beans	0·038
Speckled beans	0·038
Purple beans	0·031
Black beans	0·042
Average	0·036

From these results alone it might appear that the white variety contained the least, and the black the most, hydrocyanic acid; but this distinction does not really hold good, although it has been held that white contains the least, and purple the most, of that ingredient. Other tests we have made showed in brown beans 0·072, and in black beans only 0·029 per cent. of hydrocyanic acid, so that the generalization as to the quality of the bean according to its colour is of no value whatever.

It has been stated that the cyanogen was contained in the husk and not in the kernel of the bean, but this opinion or assumption we have proved to be quite untenable. We carefully decorticated a small specimen of black beans, and found that the husk was equal to about 10 per cent. of the whole, while the percentage of hydrocyanic acid in the decorticated portion was 0·030 per cent., and that in the husk only 0·003 per cent. We also procured a large specimen of decorticated beans, and the corresponding husks, from the mixed varieties, and found in the decorticated portion 0·061, and in the husks 0·006 per cent. of hydrocyanic acid. It is thus apparent that the kernel contains at least ten times as much hydrocyanic acid as the husk, so that the statement we have referred to is not borne out by fact. It has also been suggested that it is the small, rounded beans that contain the hydrocyanic acid, but our experience does not bear this out even in the slightest degree.

Having dealt with the Java beans, we next procured specimens of various kinds of bean and pea, and tested them for hydrocyanic acid, with the following results, which include also the average weight of the beans :

	Hydrocyanic Acid. Per Cent.	Number of Beans in 100 Grams.
English beans	none	170
Morocco beans	none	—
Smyrna beans	none	100
Java beans	0·027 to 0·137	272
Rangoon or Burma beans	0·005	368
Large haricot beans	none	64
Large haricot beans	none	78
Small haricot beans (Rangoon)	0·009	384
Small haricot beans	0·006	420
Small haricot beans	0·001	448
Small haricot beans (Chili)	none	228
Paiyin beans	none	1960
Gram	none	—
Calcutta white and gray peas	none	—
Odessa and Mutter peas	none	—

The haricot beans (both small and large) mentioned above were obtained from different sources, and were all sold for human food. The sample containing 0·001 per cent. of hydrocyanic acid was purchased by ourselves in a grocer's shop in Glasgow.

From the above table we have further and most conclusive proof that it is a mistaken idea that the white bean is free from the cyanogen compound, as the latter was present in three specimens of the white haricot bean, which is almost certain to be a cultivated variety. It will be observed that the specimen of the ordinary (or coloured) Rangoon or Burma bean given above contains only rather more than half that contained in one specimen of the small white haricot beans.

In conclusion, we may note the effect of boiling the unground beans with water, and of mashing the ground beans with warm water. In carrying out the former experiments the three samples of small haricot beans containing hydrocyanic acid were steeped overnight with water and boiled until thoroughly cooked. They were then allowed to cool, beaten up with water, and the modification of Schönbein's test applied, but hydrocyanic acid could not be detected in any of them. The hydrocyanic acid was now determined in the beans originally containing 0·009 per cent., after this treatment, and 0·002 per cent. was found. These results show clearly that a large proportion of the cyanogen compound and the whole of the enzyme have been destroyed by the boiling, but whether the amygdalin glucoside is harmless, seeing there is no enzyme present to produce hydrocyanic acid, is a question with which we cannot deal. In order to corroborate these results, Java beans containing 0·038 per cent. of hydrocyanic acid were steeped and boiled in the manner described; but after this treatment no hydrocyanic acid could be detected by the modification of the Schönbein test, although 0·019 per cent. in the form of the glucoside was actually present.

The result is entirely different when cold or warm water, as applied in the test referred to, is mixed with the ground bean. In this case we found that in two or three days practically the whole of the cyanogenetic compound was decomposed, and the cyanogen in it liberated in the form of hydrocyanic acid.

DISCUSSION.

Dr. VOELCKER remarked that Professor Dunstan drew a clear distinction between the white and the coloured beans, and spoke of the white beans as being cultivated and as containing no cyanogenetic glucoside, whereas the speckled and purple beans did contain one. He was inclined to think, however, that this was an assumption rather than the result of actual experiment, and accordingly the present paper, if the beans dealt with were botanically the same, would be of considerable value.

Mr. HENDRICK said that he had had occasion to examine a good many samples of these Java beans, and had done a little investigation on lines somewhat similar to part of that recorded in this paper. His results, generally speaking, were in accord with those of the authors. Every specimen that he had examined of these so-called Java beans, a great many thousands of tons of which had been imported into Europe during the last year or two, had yielded hydrocyanic acid in relatively large quantity. The beans varied very greatly in colour, even from bag to bag of the same consignment. In some cases white beans were more numerous than those of any other colour; in other cases the majority of the beans were black. He did not find, however, any corresponding variation in the amount of hydrocyanic acid, though, generally speaking, the darker beans contained a little more hydrocyanic acid than the light ones. He was not satisfied that the whole of the hydrocyanic acid which it was possible to produce from the beans was obtained by extracting the glucoside with alcohol, which was, he understood, the method used by the authors of this paper. In a few experiments which he had made he had found that mere soaking of the ground beans in water and subsequent distillation yielded, in every case that he had tried, a larger quantity of hydrocyanic acid than was obtained by the alcohol extraction method. He also had de-husked a sample of the beans, and had not obtained any hydrocyanic acid from the husks; it was all contained in the inner part of the beans. It had been stated that where the beans had been washed before being used there had been no poisoning. Through the kindness of certain large dealers he had been able to visit some mills where the beans were thus treated, and had seen the whole process. The washing process was a very brief one, and though it removed much dirt, it did not do more than clean the outside of the bean, and could not affect the amount of cyanogenetic glucoside present. His difficulty was to explain why there had not been far more cases of poisoning. Great quantities of the beans had gone into consumption. He personally knew of cases in which beans, of which samples from the same parcel yielded a large proportion of hydrocyanic acid, had been fed daily for weeks to cattle without ill-effect. In other cases poisoning had occurred at once, and large numbers of animals were lost. The poisoning seemed to be, so to speak, sporadic, and he felt convinced we had still something to learn as to its cause.

Mr. **Estcourt** said that he had been told by a miller near Manchester of certain beans which had poisoned a considerable number of cattle. These beans were described as "Indian mutter."

Mr. **Hendrick** said that he had examined what were known as "mutter peas," and had not found them to yield any hydrocyanic acid. It was known, however, that these peas sometimes caused poisoning of a different kind (see a paper by Dr. J. A. Voelcker, Analyst, xix., 102). In some cases the beans had been given in the form of dry meal, in some cases they had been soaked, and in some cases steamed. He had always found it quite easy to detect the hydrocyanic acid in these beans by smell, after soaking the meal in water for a short time.

Dr. **Leather** said that there was no doubt that these various beans, those from Burma especially, did contain a cyanogenetic glucoside, though it was not yet certain whether this was identical with the phaseolunatin of Professor Dunstan. It is uncertain, also, what the exact botanical nature of these beans is. They seem to be a mixture of species, but in India nearly all the crops are mixed. He thought, from what he had seen of these beans, that they probably all belong to the same genus, but it was not certain whether Professor Dunstan's statement as to *Phaseolus lunatus* is generally correct. Probably it is correct for the plant that Professor Dunstan operated on, but it is not certain that all these Burmese beans are the same. He had found a cyanogenetic glucoside in various Indian beans, and its presence seemed to be very common in leguminous plants in the East. The quantity of prussic acid obtainable varied considerably. It is very much to be desired, of course, that further knowledge should be obtained as to the physiological action of the glucoside itself. It is easy to understand the production of prussic acid by grinding up and soaking the beans in water, because these cyanogenetic glucosides are almost always associated with enzymes capable of hydrolysing them; but whether such hydrolysis would necessarily be brought about by the enzymes of the digestive organs is another question altogether, and, as far as he was aware, no work has been done in that direction. It seems very possible that the erratic effects of the beans might be explained by variations in the degree to which hydrolysis results from digestive action. In one of the first references made to him in relation to these Burmese beans, it was stated that the beans were consumed regularly by the prisoners in Burmese prisons. All that food was cooked, so that the enzyme in it was certainly destroyed; and it remained then for the enzymes of the digestive juices to hydrolyse the glucoside if they could. Apparently, as a rule, they could not. In the case of stock-feeding it is clearly necessary that such beans shall first be cooked, so as to destroy the enzyme; but it is to be recollected that the glucoside is not destroyed by a temperature of 100° C.

Mr. Hendrick asked whether Dr. Leather knew of any case of poisoning from Burma beans.

Dr. Leather said that he only knew of cases of poisoning that had occurred in this country; none had occurred in India.

Mr. Hendrick said that, as far as he knew, there had been no case of poisoning by Burma beans. It was at first stated in the newspapers that some of the beans which had caused poisoning were from Burma, but in every case, so far as he was aware, it was found on investigation that they were Java beans. The latter were different from Burma beans. Both seemed to be varieties of *Phaseolus*, though he did not know the exact species. The Burma beans were quite uniform.

Dr. Leather: No, they vary very much indeed.

Mr. Hendrick said that all the samples which he had seen of what was described as "Red Burma Beans" were red mottled beans, very similar to one another in appearance, whereas the Java beans he had seen varied greatly in colour and appearance. These red Burma beans have been imported in large quantities for many years past, and are extensively used as cattle-food in this country, but he had never heard of an authenticated case of poisoning arising from their use.

Note to Discussion by the Authors.—Our experience differs from that of Mr. Hendrick with respect to the extraction of the glucoside with alcohol, as this method gave more cyanogen than that by simply soaking the ground beans with water and distilling.

* * * * *

ON THE POLENSKE METHOD FOR THE DETECTION OF COCOANUT OIL IN BUTTER.

By S. Rideal, D.Sc., F.I.C., and H. G. Harrison, M.A., A.I.C.

(*Read at the Meeting, May* 2, 1906.)

The Polenske method for the detection of cocoanut oil in butters depends upon two conclusions at which he has arrived:

1. That in the distillation of the fatty acids, the quantity of insoluble volatile acids bears a strict relationship to the soluble volatile acids.

2. That a mixture with cocoanut oil reduces the "soluble" figure, but at the same time increases the "insoluble" figure to such an extent that each per cent. of cocoanut oil gives 0·1 c.c. $\frac{N}{10}$ alkali in excess of the figure obtained from a genuine butter giving the same "soluble" figure.

To test these conclusions a number of pure English butters have been examined, as well as butters of unknown origin. We find that the distillates from pure butters do not give the "soluble" and "insoluble" figures in such a strict ratio, but that, starting with a pure butter fat, and mixing it with cocoanut oil, the relative increase in the "insoluble" figure practically agrees with Polenske's results.

By plotting his "insoluble" figures against "soluble" a smooth curve is obtained and none of the thirty-one points lie far from the curve.

Our butters show much more divergence, but by taking an average curve in the two cases, the English butters give a curve lying considerably below Polenske's, and the curve for the unknown butters lies between the other two. Polenske's highest Reichert-Meissl value is only 30·1, while both the other series give higher values in this direction.

The following results have been obtained by us:

Pure English Butters. June and July.

County.	Number of Cows.	Pure Butter Fat.		Butter Fat + 20 per Cent. Cocoanut Oil.	
		Soluble c.c. $\frac{N}{10}$.	Insoluble c.c. $\frac{N}{10}$.	Soluble c.c. $\frac{N}{10}$.	Insoluble c.c. $\frac{N}{10}$.
Devonshire ..	?	31·2	2·05	26·7	4·0
Huntingdonshire	35	28·9	1·85	26·85	4·0
,,	4	30·95	1·7	27·15	4·0
,,	7	29·35	1·85	26·3	4·25
,,	5 or 6	30·15	1·85	26·3	4·55
,,	several	29·85	1·85	26·55	4·25
Surrey	1	31·6	1·6	—	—
Somerset	4	34·55	2·15	—	—
,, ...	?	29·0	1·7	—	—
,, ...	?	30·05	1·8	—	—
Devonshire ...	?	29·5	1·65	—	—
Somerset	12	31·25	1·95	—	—

When the first six of these were mixed with 20 per cent. of cocoanut oil giving "soluble" and "insoluble" values of 8·75 and 13·4, on the average the "soluble" figure was decreased by 3·4 and the "insoluble" figure increased by 2·3.

In order to get an idea of the changes in the values which take place under varying conditions of season, food, etc., samples have been taken at fortnightly intervals from a Huntingdonshire dairy farm since August 14, 1905. These butters are made from the milk of about thirty-five cows, a number sufficiently large to eliminate variations due to period of lactation in the individual cows. In each case the cows were milked within the week preceding the date given.

FORTNIGHTLY SAMPLES FROM SAME DAIRY.

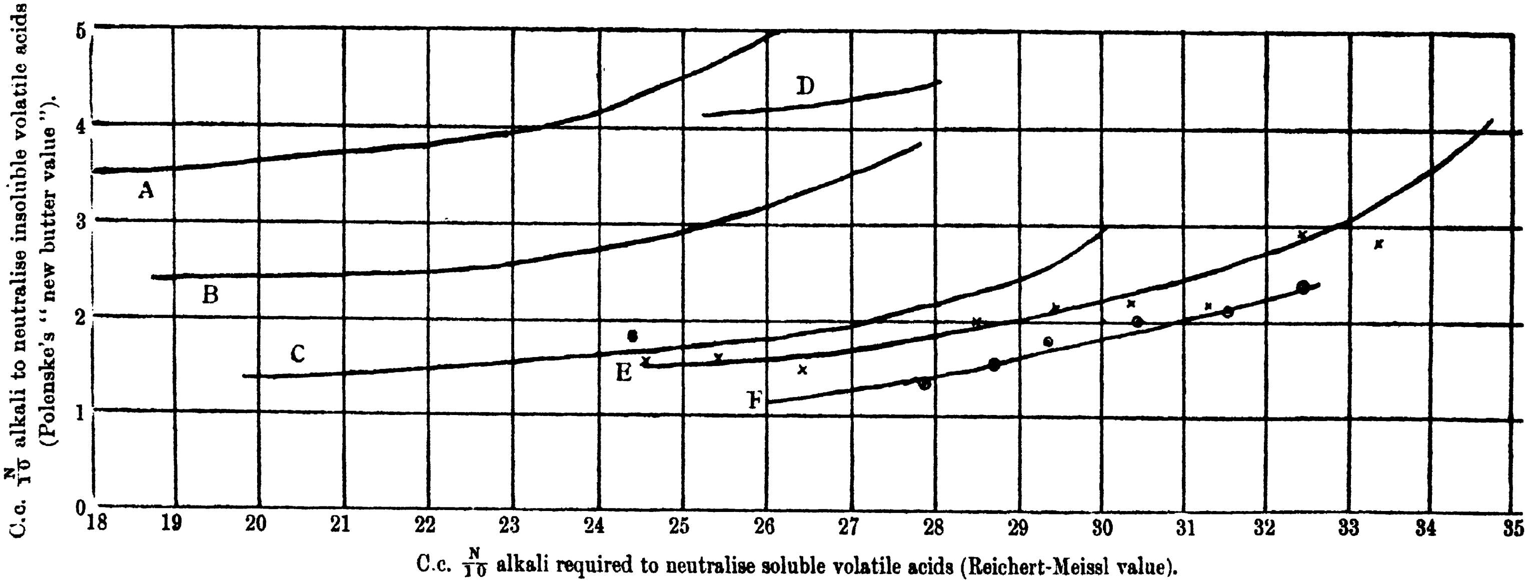
Date.	Soluble c.c. $\frac{N}{10}$.	Insoluble c.c. $\frac{N}{10}$.
June 29, 1905	28·9	1·85
Aug. 14 ,, ...	29·55	1·7
,, 28 ,, ...	28·5	1·2
Sept. 11 ,, ...	27·95	1·3
,, 25 ,, ...	27·8	1·35
Oct. 9 ,, ...	29·2	1·35
,, 23 ,, ...	32·1	2·3
Nov. 6 ,, ...	30·7	2·2
,, 20 ,, ...	32·9	2·05
Dec. 4 ,, ...	30·1	1·75
,, 18 ,, ...	31·75	2·2
Jan. 1, 1906	30·5	2·1
,, 15 ,, ...	30·25	2·25
,, 29 ,, ...	31·8	2·1
Feb. 12 ,, ...	32·3	2·75
,, 26 ,, ...	31·25	2·3
Mar. 12 ,, ...	31·8	2·5
,, 26 ,, ...	29·05	2·3
April 9 ,, ...	30·8	2·4

Until October the cows were entirely on grass and out all the time, then they began to come in at night and have linseed and cotton-cake in addition. During November and December the cows were kept under shelter, and in addition to cake were fed with hay, oatmeal, and rabi. After December mangel-wurzel was substituted for the rabi, and some of the cows began to go out for part of the day.

The average results obtained from all these English butters are given in the curve. They were obtained by taking averages of the results lying within a range of 1 c.c. of soluble volatile acids as given in the following table :

AVERAGE RESULTS FOR PURE ENGLISH BUTTERS.

No. of Samples.	R. M. Values.	Average.	
		Soluble c.c.	Insoluble c.c.
2	27–28	27·88	1·33
2	28–29	28·70	1·53
7	29–30	29·36	1·77
8	30–31	30·44	2·00
7	31–32	31·52	2·10
3	32–33	32·43	2·37
0	33–34	—	—
1	34–35	34·55	2·15



A, Polenske's curve for 80 per cent. butter-fat +20 per cent. cocoanut oil.
B, ,, 90 ,, ,, +10 per cent. ,, (average).
C, ,, pure butters.
D, English butter +20 per cent. cocoanut oil.
E, Unknown butters.
F, Pure English butters.

⊙ Average English butters.
× Average unknown butters.

In a similar manner the figures obtained from one hundred and three unknown butters, presumably genuine, were averaged as follows:

AVERAGE RESULTS FOR 103 UNKNOWN BUTTERS.

No. of Samples.	R. M. Values.	Average.	
		Soluble c.c.	Insoluble c.c.
3	24–25	24·58	1·55
4	25–26	25·46	1·58
7	26–27	26·42	1·46
8	27–28	27·27	1·73
19	28–29	28·50	1·98
29	29–30	29·45	2·12
20	30–31	30·36	2·16
6	31–32	31·30	2·13
4	32–33	32·41	2·91
2	33–34	33·35	2·83
1	34–35	34·7	4·1

During the same period margarines were examined in the same way, but in no case gave an "insoluble" value greater than 1 c.c. $\frac{N}{10}$ alkali; and mixtures made with butter, margarine, and cocoanut oil gave figures which indicated the amount of cocoanut oil present, but no indication in themselves of margarine if a butter of high Reichert value were taken.

Cheese fats examined by the same method, while giving, as is well known, the Reichert value higher than is general with butters, gave "insoluble" values which are in the same ratio to the "soluble" values as in the case of butters.

DISCUSSION.

The PRESIDENT (Mr. Bevan) asked whether the saponification was done with glycerin or with alcohol. The Polenske number, as was well known, differed considerably according to whether glycerin or alcohol was used, being, if he remembered rightly, higher with glycerin than with alcohol.

Mr. HARRISON said that glycerin had been used, following the process as prescribed.

Mr. E. R. BOLTON said that he had been particularly interested in this paper in so far as it related to cocoanut oil, with which he had had more experience than with butter. The results he had obtained were more or less in agreement with those now given, except that he usually found a higher insoluble acids figure, and a lower Reichert-Meissl value than the authors had. But he had obtained a Reichert-Meissl figure as high as 9 for cocoanut oil, whereas the highest mentioned by Polenske was 7·7. He had also noticed that, as a rule, the figure for insoluble volatile acids in cocoanut oil was from 2·2 to 2·5 times the Reichert-Meissl value. It was difficult, however, to get samples of cocoanut oil that were perfectly normal. In many cases the "stearine" might have been removed, and some samples might contain palm kernel oil. It was, perhaps, worth noticing that palm kernel oil gave an average Reichert-Meissl figure of 5, the figure for insoluble volatile acids being 10. As far as the effect of different modes of feeding was concerned, the Polenske numbers seemed to come out very regularly. He did not know whether Polenske had made any notes as to the feeding of the cows in his investigations, but it was a matter of common experience that the use of cake did affect to a certain extent the composition of the butter-fat, and certainly cocoanut cake had some effect in many cases.

Mr. HINKS inquired whether in any of these cases the ordinary Reichert-Wollny figure, as well as the Reichert-Meissl figure, had been determined in conjunction with the Polenske number. If so, it would be interesting to see what difference was made by the use of glycerin and by the more rapid distillation.

Mr. HEHNER said that his experience was quite in agreement with that of the authors, that the relationship between the Polenske number and the Reichert-Wollny number was not in every case so definite that one could safely use it as the basis of a definite charge of adulteration. Broadly speaking, of course, it was agreed that the lower the Reichert-Wollny figure, the lower would be the proportion of insoluble volatile acids. Owing, however, to the varying circumstances under which butter was produced, one could not be guided by broad facts in particular cases. His own experience over some considerable time had been that the Polenske numbers shown by pure butter were about the same as those which the authors had obtained, and it would be seen that the difference between the authors' curve for English butters and Polenske's curve for pure butter-fat would allow a margin of about 10 per cent. for admixture. Then came the question of the state of the insoluble volatile acids. At one time it was thought that fluidity of the insoluble volatile acids indicated the presence of cocoanut oil, and that to a certain extent was true, but it was not universally true. In the case of pure butter showing a low Reichert-Wollny figure the insoluble volatile acids were almost invariably solid. When a low Reichert-Wollny figure was associated with fluid insoluble volatile acids, and especially when the Polenske number exceeded 2, the proof of the presence of cocoanut oil was fairly definite. But in butters showing Reichert-Wollny figures of 25 or over, the insoluble volatile acids were in many cases quite fluid. He did not think it safe to rely upon any single figure, however important it might be in itself. Cocoanut oil, as was well known, had a very low iodine value and a very low refraction, whereas genuine butter with a low Reichert-Wollny figure had almost invariably a high refraction and a high iodine value. In this connection micro-polariscopic examination was of some importance. It was, however, beset by many pitfalls, and used alone would be worth nothing. In the process suggested by Knud Jensen and Kirschner unfortunately the silver caprylate was too soluble, and the solubility too much influenced by temperature and time allowed for the precipitation, to lead to reliable figures.

Mr. ESTCOURT said that, from experiments with butter made in his own laboratory, in connection with some samples that were suspected of containing cocoanut oil, he had come to the conclusion that in genuine butter having Reichert number less than 29 the figure for insoluble volatile acids was rarely over 2.

Mr. HARRISON, in reply, said that it must be agreed that between the figures they found and those of Polenske there was, as Mr. Hehner had said, an appreciable margin of possible adulteration. Polenske himself stated that nothing under 10 per cent. could be detected—that, of course, being the minimum, and each 1 c.c. of increase being taken as equivalent to 10 per cent. of cocoanut oil. Towards the end of the curve, however, there was a wider range of variation still. There was, therefore, considerable difficulty in making the process quantitative, apart from the question of the actual detection of cocoanut oil. If, however, they could satisfy themselves that this process was capable of detecting the presence of cocoanut oil with certainty, it might be possible to arrive at some approximate estimate of the quantity. With regard to saponification, although the authors had only used glycerin, he should think that, as a rule, with alcohol a slightly higher Reichert-Meissl value would be obtained than with glycerin. The difference, however, would not be very great. He knew nothing about the Polenske value under these conditions.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Proteolysis in Cow's Milk preserved with Formaldehyde. G. Tice and H. C. Sherman. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 189-194.)—Analyses of samples of milk preserved with formaldehyde show that very considerable proteolysis takes place after some time, the albumin being largely digested before the amount of casein is appreciably reduced, though as digestion proceeds and the casein diminishes the proportion of nitrogen existing as albumin and syntonin changes but little, the increase appearing first in the proteoses, then in the peptones and amino-compounds. Only slightly more nitrogen was precipitated by phosphotungstic acid than by tannin, indicating the presence of only small amounts of diamino-acids. Relatively large amounts of proteoses have been found at a rather advanced stage in the digestion, a result entirely different from that of bacterial digestion of milk. In one case, while 95 per cent. of the casein had been digested, less than 4 per cent. of the original lactose was lost, as compared with milks preserved with sodium fluoride or salicylate or hydrogen peroxide, in which, after 25 to 30 per cent. of the lactose had been destroyed, no marked digestion of the casein was noticed, proving the effect of an added antiseptic in determining the character of the fermentation subsequently undergone.

W. H. S.

The Determination of Proteids in Milk. Trillat and Sauton. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 205-207.)—The method is based upon the fact that formaldehyde renders the proteids of milk insoluble. Five c.c. of the milk are diluted to 25 c.c. with water, and the liquid boiled for five minutes, then treated with 5 c.c. of commercial formalin, boiled for two or three minutes longer, and allowed to stand for five minutes. It is next shaken with 5 c.c. of a 1 per cent. solution of acetic acid and the precipitate collected on a weighed filter, which is washed with water, and subsequently extracted with acetone to remove the fat. Finally, it is dried at 75° to 80° C. and weighed. The following results in grams per litre of milk were thus obtained :

	Proteid.		Proteid.
Cow's milk	39·11	Goat's milk	36·64
Ditto, with 10 per cent. of water	34·33	Ass's milk	21·03
Ditto, with 50 per cent. of water	19·50	Colostral milk (cow)	11·00
Sheep's milk	55·52	Whey	4·50

Experiments are described to show that the proteid is completely separated (the amount of nitrogen in the precipitate is identical with that in the milk), and that the elementary composition of the precipitate agrees with that of casein. The author also concludes that the weight of the proteid is not increased by the action of the formaldehyde, for he finds that casein rendered insoluble by being left under a bell-jar containing traces of trioxymethylene does not alter appreciably in weight, whilst, on the other hand, the strength of a formalin solution is not changed appreciably after precipitating the casein, and the weight of proteid separated from the same milk in different determinations with varying amounts of formaldehyde remains the same. Sterilization of the milk has no influence on the accuracy of the results, and the method is applicable to milk preserved with potassium bichromate.

C. A. M.

On the Use of the "Silver Value" for the Detection of Cocoanut Oil in Butter. F. Jean. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 121-124.)—The method devised by Wijsman and Reijst (ANALYST, xxxi., 158) has been tried by the author upon twelve samples of doubtful butter, and as the second silver value was invariably lower than the first, the conclusion arrived at was that the butters were abnormal, but free from cocoanut oil. On the other hand, the method of Muntz and Coudon (ANALYST, xxx., 155) gave ratios of 22 to 36, which, in conjunction with the other analytical values, pointed to the presence of cocoanut oil. Thus the following results were obtained with two of the samples :

	Oleo refractometer Reading.	Saponification Value.	Reichert-Meissl Value.	First Silver Value.	Second Silver Value.	Muntz and Coudon's Method.			
						Soluble Acids.	Insoluble Acids.	Ratio × 100.	Polariscope.
Butter, No. 1	35	228	27·2	5·06	4·81	4·81	1·76	36	Abnormal
,, No. 3	35	229	26·2	4·95	4·50	4·50	1·14	26	Abnormal

In order to determine the cause of these contradictory results, the author prepared mixtures of pure butter with 10 per cent. of a cocoanut-oil mixture in common use for adulterating purposes, and with 10 per cent. of a mixture in equal parts of cocoanut oil, karité, butter, and lard, and obtained the following results:

	Oleo-refractometer Reading.	Saponification Value.	Reichert-Meissl Value.	Soluble Volatile Acids.	Insoluble Volatile Acids.	Ratio.	First Silver Value.	Second Silver Value.
Adulterated Butter, No. 1	32·0	220	26·4	4·72	2·29	48	6·18	5·80
,, ,, No. 2	32·5	216	26·7	4·69	1·45	30	6·16	5·52

The presence of the cocoanut oil (3·4 and 2·5 per cent. respectively) is here plainly shown by the ratio in Muntz and Coudon's method, whereas from the silver values both samples would have been judged to be pure. Hence the author concludes that the method of Wijsman and Reijst can only be of value as a confirmative test, and that when it gives negative results cocoanut oil in admixture with other fats may still be present.

C. A. M.

The Characteristics of Karité Butter. F. Jean. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 201-203.)—Karité or fulwar butter is extracted from the seeds of *Bassia butyracea*, a tree growing in the Himalayas and in the tropical zone of Africa. According to the author, large quantities of the seeds and of the fat are sent to England, the latter being used in the adulteration of butter, lard, and cocoa-butter. The kernels examined by the author had the following composition: Water, 10·05; fat, 35·49; soluble extractives, 26·44; tannin, 3·2; ash, 2·50; and cellulose, 22·52 per cent. The butter is a white, paste-like fat, with a slight odour and an astringent taste. It can be purified by a method similar to that used in deodorizing cocoanut oil for food purposes. The following analytical values were obtained with a sample of the fat extracted with petroleum spirit and of fat prepared by the natives by crushing the nuts with water:

Fat.	Oleorefractometer at 45° C.	Melting-point. °C.	Acid Value.	Saponification Value.	Iodine Value.	Reichert-Meissl Value.	Soluble Fatty Acids.	Hehner Value.
Extracted in laboratory	+22°	30	—	175–176	—	2·6	—	—
As imported	+18°	30	9	175	19·75	1·19	trace	91·2

The fat consisted of 69·28 per cent. of solid fatty acids, 21·92 per cent. of liquid acids, and 8·85 per cent. of glycerin. The fatty acids, separated by Renard's method, melted at 67·8° C. Neither caproic nor caprylic acid was present in the butter.

C. A. M.

The Action of Flour, etc., on Hydrogen Peroxide. W. Bremer. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 569-577.)—The author's experiments show that the method proposed by Wender (ANALYST, 1906, xxxi., 73) for the valuation of flours is at present of little use. The evolution of oxygen is erratic, equal weights of the same flour yielding varying volumes of gas, and the quantity is not proportional to the amount of flour employed, or to the percentage of bran in the flour. The power of decomposing hydrogen peroxide is diminished if the flour be heated previously to a temperature of 98° C., a moist heat having more effect than a dry heat. As both the aqueous extract and the insoluble residue, obtained on extracting bran with water, decompose hydrogen peroxide, the assumption is raised that more than one constituent of the bran is capable of causing the decomposition. The presence of mercuric chloride or of hydrochloric acid inhibits the reaction, as does also that of alcohol. Whilst well-washed gluten decomposes hydrogen peroxide to a slight extent, no action was noticed in the case of aqueous solutions of diastase.

W. P. S.

Formic Acid as a Preservative of Lemon-Juice. Von Küttner and Ulrich. (*Zeit. öffentl. Chem.*, 1906, xii., 201-207.)—The deterioration of fresh lemon-juice due to the formation of acetic acid may be prevented by the addition of from 0·1 to 0·4 per cent. of formic acid. The addition, of course, increases the acidity of the juice, and the results of experiments are given which show that the formic acid distils over quantitatively with the other volatile acids present in the juice. W. P. S.

Composition of Tomatoes and Tomato-Juice. W. Stüber. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 578-581.)—The following results were obtained on the analysis of two samples of tomatoes and their respective juices:

	Fruit, 1.	Fruit, 2.	Juice, 1.	Juice, 2.	Juice, 3.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water	94·52	95·13	96·00	96·19	95·48
Nitrogen	0·116	0·159	0·098	0·088	0·128
Ash	0·50	0·63	0·50	0·63	0·65
Alkalinity of ash (c.c. $\frac{N}{1}$ acid) ...	4·62	5·50	5·20	6·00	6·40
Petroleum spirit extract ...	0·06	0·07	—	—	—
Sugars (after inversion)	2·51	3·21	2·34	1·96	2·53
Free acid (as citric) ...	0·41	0·48	0·60	0·61	0·46
Phosphoric acid (P_2O_5) ...	0·044	0·059	0·031	0·039	0·025

The sample of juice No. 3 was obtained from Canadian tomatoes, the other samples being of German origin. Tartaric, malic, and succinic acids could not be detected in any of the samples. W. P. S.

Determination of Talc on Rice, Pearl-Barley, etc. R. Kržižan. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 641-650.)—The quantity of talc present as a coating on rice, pearl-barley, or other grain can only be correctly determined by directly weighing the mineral after detaching it from the grains. As the mineral is readily attacked by hydrochloric acid, especially after ignition, the method proposed by Matthes and Müller (ANALYST, 1905, xxx., 206) is untrustworthy, and methods based on the determination of the magnesia in the ash of the sample (the magnesia being subsequently calculated into talc) give incorrect results, owing to the varying composition of the mineral itself, and to the magnesia natural to the grains. The author, therefore, employs a process in which a weighed portion of the sample is heated with a little hydrogen peroxide and dilute ammonia; bubbles of gas form between the grain and its coating, and completely loosen and remove the latter. The turbid liquid is decanted, the grains are shaken and washed several times with water, and the total liquid then rendered acid with hydrochloric acid, and the organic matter oxidized by the addition of a few grams of chromic acid. The mixture is heated to boiling, filtered, and the residue is washed with water and ignited at the lowest possible temperature. The weight of residue obtained represents the amount of talc in the portion of the sample taken. Besides talc, other minerals, such as mica and certain clays, are used for coating grains. (*Cf.* "Facing of Rice," this vol., p. 40.) W. P. S.

The Determination of Alcohol in Chloroform. M. Nicloux. (*Bull. Soc. Chim.*, 1906, xxxv., 330-335.)—A small proportion of alcohol is commonly added to chloroform as a preservative. It can be separated by shaking 5 c.c. of the sample with 20 c.c. of water, which extracts the whole of it, and it can then be determined in the supernatant aqueous layers by the author's bichromate method. Five c.c. of this alcoholic solution (containing not more than 1 part of alcohol in 500) are mixed in a test-tube with 0·1 to 0·2 c.c. of a solution of potassium bichromate (19 grams per litre), and then with pure sulphuric acid (66° B.), which, when added in sufficient quantity (4·5 to 6 c.c.) decolorizes the solution. The liquid is now gradually titrated with the bichromate solution, being shaken and gently heated after every addition, until the colour changes from greenish-blue to a permanen greenish-yellow. The change is most readily recognised in solutions containing les than 0·2 per cent. of alcohol. It is advisable to make a second determination in which the amount of bichromate used in the first experiment, less 0·1 c.c., is run in at once, the sulphuric acid added, and the liquid boiled for a moment. The conten of the tube ought to remain greenish-blue, whilst in another determination in which 0·1 c.c. more than the first reading is added, the colour should change to greenish-yellow. The number of c.c. consumed, divided by 1,000, gives the amount of absolute alcohol in c.c. per c.c. of solution. When the proportion of alcohol is less than 0·1 per cent. it is best to double the strength of the bichromate solution. Six samples of chloroform examined by this method were found to contain from 1·4 to 10·4 c.c. of absolute alcohol per litre. Test experiments with chloroform containing known quantities of alcohol showed that the results were concordant and accurate.

C. A. M.

TOXICOLOGICAL ANALYSIS.

A Colorimetric Method of Determining Morphine. L. Georges. (*Journ. Pharm. Chim.*, 1906, xxiii., 513-516.)—The solution of morphine, extracted by the Stas-Otto method, gives a yellow or yellowish-red coloration on treatment with iodic acid, and the colour changes to yellowish-brown on the addition of ammonia. In each case the intensity of the colour is proportional to the amount of morphine, and can be matched with the shade produced by a standard solution of morphine (1·256 grams of morphine hydrochloride per litre = 0·001 gram of morphine per c.c.). In making a determination, 5, 10, or 20 c.c. of the solution of unknown strength and the same quantity of the standard alkaloid solution are treated simultaneously with 5 c.c. of a 5 per cent. solution of iodic acid, and the colours compared, before and after the addition of 5 c.c. of a 10 per cent. solution of ammonia. The colour produced by the iodic acid alone reaches its maximum intensity after half a minute, and begins to fade after fifteen minutes. In the case of the ammoniacal coloration the maximum is only reached after two or three minutes. The author states that this method has invariably yielded good results in his hands, but points out that the reduction of iodic acid cannot be regarded as an absolute test of the identity of morphine extracted by Stas's method. C. A. M.

On the Quantitative Toxic Properties of Carbolic Acid as compared with those of other Poisons. Th. Bokorny. (*Chem. Ztg.*, 1906, xxx., 554.)—The author points out that in order to estimate correctly the toxic properties of any substance, it is necessary to ascertain not only the degree of dilution at which it will still act, but also the total minimum quantity necessary, and complains that this fact is commonly lost sight of in the case of antiseptics. He has determined the "lethal dose" for a number of poisons acting on 10 grams of fresh brewers' yeast (containing 30 per cent. of dry substance), and communicates his results, together with some previously published, as follows: Carbolic acid, between 0·05 and 0·1 gram; formaldehyde, 0·02 to 0·04; acetaldehyde, above 0·5; *o*-oxybenzaldehyde, 0·25 to 0·5; acetic acid, 0·2 to 0·4; copper sulphate, 0·001 to 0·0025; corrosive sublimate, 0·005 to 0·01; sodium fluoride, 0·05 to 0·1; hydrofluoric acid, 0·01 to 0·025; silver nitrate, 0·01 to 0·02; zinc sulphate, 0·05 to 0·1; lead acetate, 0·05 to 0·1; hydrochloric acid, 0·05 to 0·1; caustic soda, 0·05 to 0·1; potassium permanganate, 0·02 to 0·05; chlorine, 0·015 to 0·03; tannic acid, 0·5 to 1. A. G. L.

ORGANIC ANALYSIS.

Analysis of Sugar Mixtures. C. A. Browne. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 439-453.)—Since the weights of two different sugars reducing the same amount of copper bear a constant ratio to one another, the differences in the reducing powers of the various sugars can be very simply expressed by determining this ratio for each sugar, compared with dextrose as a standard. These constants are termed the "dextrose ratios" or "equivalents," and based on the conclusions that (1) the dextrose equivalent of a mixture of reducing sugars is equal to the sum of the dextrose equivalents of the individual sugars (which the author has proved, notwithstanding statements to the contrary); and (2) the polarization of a mixture of sugars is equal to the sum of the polarizations of the individual sugars present, formulæ are developed which enable the percentage composition of a mixture of any two sugars, or of three sugars, one of which is sucrose, to be ascertained from determinations of the copper-reducing power of the mixture and of its polarization. Two sources of error arise in such separations, due to (1) the slight reducing action of sucrose upon Fehling's solution, and (2) the change in rotation of lævulose in neutral and acid solution. In some experiments with a mixture of dextrose and sucrose, the error due to the first-mentioned cause is found to be inversely proportional to the amount of dextrose present, and the following correction gives concordant results: Divide the grams of sucrose in the 25 c.c. of solution to be analysed by Allihn's method by the mgms. of dextrose found +40, and subtract the quotient in grams from the dextrose found. The correction for the second source of error depends upon the concentration of the acid used for inversion and the dilution of the sugar solution; for 10 c.c. fuming HCl (specific gravity 1·18) and 100 c.c. of sugar solution the correction is 0·036 per cent. for each percentage of lævulose. Experiments on a number of known mixtures of various sugars have proved the accuracy of the method.

W. H. S.

On the Colour Reactions of Sesame Oil. P. Soltsien. (*Chem. Rev. Fett. u. Harz-Ind.*, 1906, xiii., 138.)—Further experiments have confirmed the author's conclusion that the furfural reaction and the tin reaction of sesame oil are not due to one and the same substance. Both compounds can be extracted by shaking the oil with alcohol of 90 per cent. strength, but thorough extraction with hydrochloric acid of specific gravity 1·125 removes the substance that gives the furfural reaction, whilst the oil still gives the tin reaction as strongly as before. The tin reaction is also quite independent of Bishop's reaction. Hence, if a sesame oil for any reason gives neither the furfural nor Bishop's reaction, it may still be possible to identify it by the tin reaction (see ANALYST, xxvii., 363; xxviii., 298, 364).

C. A. M.

On Indian Ghedda Wax. Georg Buchrer. (*Chem. Zeit.*, 1906, xxx., 528.)—The author takes exception to the statement of Dr. J. König ("Die Untersuchung landwirtschaftlich und gewerblich wichtiger Stoffe," ed. 1906, p. 939), that the Ghedda wax of India is not to be regarded as real beeswax. The matter is of importance, since for church use only candles made from beeswax may be used. Ghedda wax is produced by three species of bees, two of which—*Apis dorsata* and *Apis florea*—stand evolutionally directly before the European *Apis mellifica*, whilst the third—*Apis indica* or *fasciata*—appears to be a variety of *Apis mellifica*. Ghedda wax is qualitatively of the same composition as ordinary beeswax, but differs from it quantitatively, the amount of free cerotic acid being very much less and the quantity of esters very much higher in the Indian wax. This is shown in the following table:

Source.		Melting-point, 0° C.	Acid Number.	Saponification Value.	Iodine Value (Hübl).
Apis dorsata ...	Average	63·1	7·0	96·2	6·7
(23 samples)	Maximum	67·0	10·2	105·0	9·9
	Minimum	60·0	4·4	75·6	4·8
Apis florea ...	Average	64·2	7·5	103·2	8·0
(5 samples) ...	Maximum	68·0	8·9	130·5	11·4
	Minimum	63·0	6·1	88·5	6·6
Apis indica ...	Average	63·25	6·8	96·2	7·4
(7 samples)	Maximum	64·0	8·8	102·5	9·2
	Minimum	62·0	5·0	90·0	5·3
Apis mellifica ...	Average	63·25	20·0	95·0	7·5
	Maximum	65·0	21·0	99·0	11·0
	Minimum	61·5	17·50	87·5	4·0

The author is of opinion that Ghedda wax is to be regarded as a true beeswax, and as such admissible for church use. A. G. L.

The Reaction of Mercuric Acetate with Camphene and Pinene. L. Balbiano. (*Reale Accad. di Lincei;* through *Pharm. Journ.*, 1906, vol. 76, p. 641.)—When equal volumes of benzene and an essential oil containing camphene are shaken with a 25 per cent. solution of mercuric acetate, and allowed to stand for thirty days, a crystalline compound, $C_{10}H_{16}O(HgC_2H_3O_2)_2$, is formed which is insoluble in water, alcohol, and ether. When the crystals are suspended in water and treated with a current of hydrogen sulphide they regenerate camphene. When pinene is subjected to prolonged contact with aqueous mercuric acetate solution, the reaction is quite different, Δ^6 oxymenthenone being formed. W. P. S.

A New Method for the Determination of Nitrogen in Nitrocellulose. M. Busch. (*Chem. Ztg.*, 1906, xxx., 596.)—The nitrocellulose is saponified with caustic alkali in the presence of hydrogen peroxide, which prevents reduction of the nitrate by the cellulose further than to nitrite. On acidifying in the presence of hydrogen peroxide, the nitrite is also quantitatively oxidized to nitrate. The determination is then completed by adding "nitron," and weighing the nitron nitrate ($C_{20}H_{16}N_4{\cdot}HNO_3$). A. G. L.

Extraction of Tanning Materials for Analysis. F. P. Veitch and H. H. Hurt. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 505-512.)—Further experiments with the continuous extractor confirm the conclusion that this extractor gives the most complete extraction, all soluble constituents being removed by it in larger quantities than by other extractors. It has been stated that with sumac and canaigre the temperature must be kept at from 50° to 60° C. until at least half the volume of the extract is obtained; but these materials were extracted satisfactorily in the continuous extractor with from 200 to 300 c.c. of water below 100° C., the extraction being completed at steam heat. All materials should be ground to pass at most a millimetre sieve, and extraction should be continued for at least sixteen hours, preferably for twenty-four hours. W. H. S.

Strychnine Tannate and its Application to the Analysis of Tanning Materials. S. R. Trotman and J. E. Hackford. (*Ledermarkt, Collegium*, 1906, 69; through *Chem. Zeit.*, 1906, xxx., *Rep.* 167.)—The tannate of strychnine is almost insoluble in water, whilst the gallic acid compound is easily soluble. In making an analysis, the tanning material is extracted with alcohol. The extract, which should contain about 0·5 gram of active substance, is evaporated to 50 c.c., and made up to 100 c.c. with water. The liquid is filtered from the resinous bodies, and 25 c.c. of the filtrate are diluted with water, after which 0·25 gram of strychnine dissolved in 50 c.c. of alcohol and 50 c.c. of water is added, and the whole diluted to 250 c.c. with water. The precipitate is filtered off on to a Gooch crucible and dried *in vacuo*. Experiments with pure tannin showed that if an excess of strychnine is present, the tannate contains 1 molecule of strychnine for every 1 molecule of tannic acid. A. G. L.

The Determination of Uric Acid in Urine. G. Guérin. (*Journ. Pharm. Chim.*, 1906, xxiii., 516, 517.)—One gram of anhydrous sodium carbonate is dissolved in 120 to 125 of the urine and the resulting precipitate of phosphates separated by filtration. One hundred c.c. of the filtrate are mixed with 25 c.c. of a solution of ammonium nitrate (50 grams in 100 c.c.), followed by 5 c.c. of ammonia solution, and allowed to stand until the next day, when the uric acid will have been quantitatively precipitated as ammonium urate. The precipitate is collected, washed with a solution containing 10 per cent. of ammonium nitrate and 1 per cent. of ammonia, and transferred with the aid of a jet of water into an Erlenmeyer flask. The turbid liquid (about 100 c.c.) is mixed with 40 c.c. of sulphuric acid (1 : 1), heated to 50° C., and titrated with a standard solution of potassium permanganate (1·5 gram per litre), the flask being shaken after each addition. The number of c.c. consumed multiplied by 0·00356 gives the amount of uric acid in 100 c.c. of the urine. The method is directly applicable to urines containing albumin. Urines that give a deposit of urate ought to be slightly heated on the water-bath and shaken until the sediment dissolves, before the addition of the sodium carbonate. C. A. M.

Estimation of Ammonia in Used Lime Liquors. H. R. Procter and D. McCandlish. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 254-256.)—A current of air, freed from carbon dioxide and ammonia by passage through U-tubes filled with NaOH and H_2SO_4, is rapidly drawn, by means of a water-pump, through the ammoniacal liquor contained in a "glass worm" immersed in a water-bath at 90° C., and thence through a U-tube containing a known volume of standard acid. Frothing is obviated, and the time required reduced from one hour to thirty minutes by replacing the "glass worm" by the following apparatus: A piece of glass tubing, A (about $\frac{3}{16}$-inch bore), is drawn out to a fine point at one end, which is bent up as shown in the figure. Over this point is fixed, by wiring round a piece of cork, a second tube, B, of the same diameter as A, but slightly widened at the end. The tubes are then fitted in a wide test-tube (10 inches by 2 inches) containing sufficient water to cover their junction, the long arm of A passing through a two-holed rubber stopper, and being connected with U-tubes containing NaOH and H_2SO_4 for purifying the air. Through the other hole of the stopper is a short piece of bent glass tubing making connection with a U-tube containing 30 to 40 c.c. of $\frac{N}{50}$ acid, which in turn is attached to the pump. The tubes A and B are so adjusted that the passage of the air causes a continuous stream of water up to B, air also escaping from the bottom of B and rising outside it. The test-tube is filled with broken glass up to within 2 inches of its top, and over the end of B is placed the inverted head of a thistle funnel, the aperture where the stem was connected having been carefully sealed. The tube is then placed in a water-bath at 90° C., 50 c.c. of the lime liquor introduced into A through a filter funnel, and slight suction applied, whereby the liquor is continuously raised to the top of B and thrown back by the thistle funnel upon the broken glass, whence it runs back to the bottom of the tube, the ammonia being thus completely drawn off within thirty minutes.

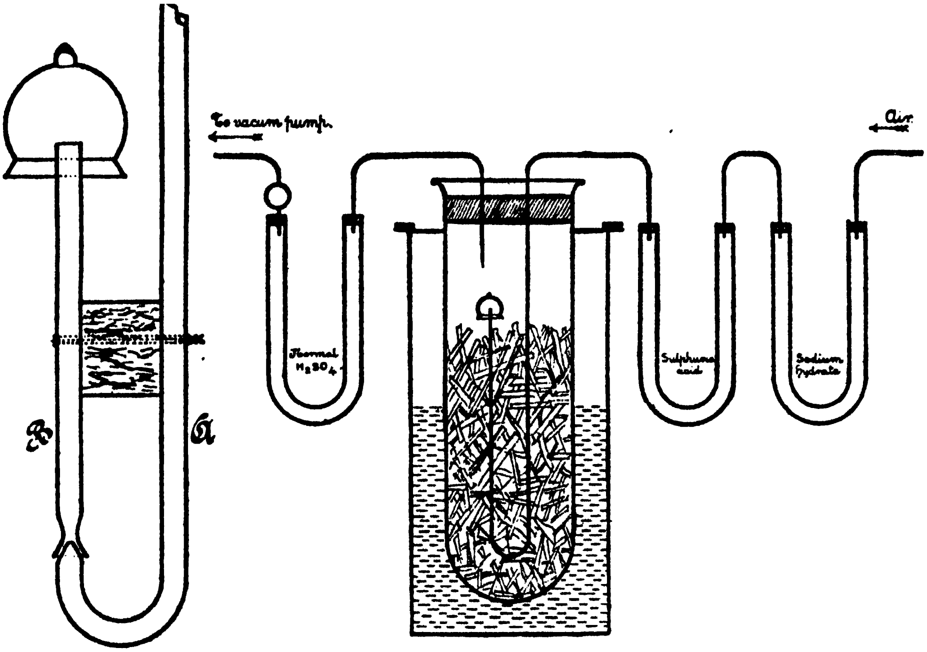


FIG. 1.

W. H. S.

Notes on the Determination of Ash in Elementary Organic Analysis. Fritz von Konek. (*Chem. Ztg.*, 1906, xxx., 567.)—The author finds that in analysing coals the amount of ash left in the carbon and hydrogen determination (carried out in an electrically-heated furnace) is always higher than that given by the usual method of ashing the sample in a platinum crucible in the air. He shows that platinum boats increase in weight (up to 0·8 mgm.) on ignition in oxygen, coming back to their original weight on prolonged heating in air. Many substances (calcium carbonate, iron oxides, etc.) also behave differently according as to whether ignited in oxygen or air. Finally, the quantity taken for the elementary analysis is so small that it is very difficult to obtain a fair sample as regards ash. For all these reasons the author prefers to take the usual ash figure obtained by burning 1 or 2 grams of the fuel in a crucible. A. G. L.

Combustion of Halogen Compounds in Presence of Copper Oxide. C. J. Robinson. (*Amer. Chem. Journ.*, 1906, vol. 35, pp. 531-533.)—The need for a separate combustion tube filled with lead chromate is obviated by placing just in front of the boat in the ordinary combustion tube a cartridge of heavy copper wire gauze rolled into the form of a hollow cylinder, 6 or 7. cm. long, and filled with pure lead chromate, the ends being closed by wrapping with Cu wire, which, passing also lengthwise through the cartridge, is looped at each end to facilitate removal from the tube. The arrangement has given good results for the determination of C, H, and N in halogen compounds, and would probably also be suitable for the estimation of C and H in sulphur compounds. W. H. S.

INORGANIC ANALYSIS.

Sampling of Gold Alloys. E. A. Smith. (*Chem. News*, 1906, 93, pp. 225, 226.)—Analyses are given proving the importance of carefully removing the coloured surface from gold alloys of all standards before taking the sample for assay, whether by cutting or scraping, the error due to inclusion of the coloured surface amounting with cut samples to as much as 1·27 per cent., and with scraped samples to 13·57 per cent. W. H. S.

The Detection of Gold and Platinum in Inorganic Analysis. J. Petersen. (*Zeit. anal. Chem.*, 1906, xliv., 342-344.)—The solution of the metals, which should be slightly acid, is treated with an excess of zinc turnings, which precipitate mercury, silver, lead, bismuth, copper, cadmium, platinum, gold, arsenic (part escapes as arseniuretted hydrogen), antimony, and tin, with some cobalt and nickel, in a granular or spongy form. The contents of the vessel are gently heated for about fifteen minutes and filtered, and the filtrate examined as in the case of the usual filtrate from the hydrogen sulphide precipitation. The precipitate is washed and heated with dilute hydrochloric acid, which dissolves the excess of zinc, the cadmium and tin, and some cobalt. The residue is washed until the washings give no reaction with silver nitrate, and is then boiled with dilute nitric acid. This dissolves the mercury, lead, copper, bismuth, and nickel, leaving a residue of gold, platinum, antimony, and antimonic acid. This residue is washed, dried, and ignited in a porcelain crucible with 1 to 2 parts of ammonium nitrate, and 5 parts of ammonium chloride, with the result that the antimony is volatilized as chloride, and a residue of unaltered gold and platinum obtained. They are dissolved in *aqua regia* and the solution divided into two parts, one of which is tested for platinum by means of ammonium chloride, and the other for gold by means of sulphurous acid or an alkaline solution of hydrogen peroxide. The filtrates are united, and after evaporation of the free acid, the other metals are precipitated by means of hydrogen sulphide. It is stated that platinum and gold were readily detected by this method in 50 c.c. of a solution containing 0·04 per cent. of the former and 0·01 per cent. of the latter, with about 0·5 per cent. of each of the other metals. C. A. M.

The Action of Sulphuric Acid on Platinum. M. Quennessen. (*Chem. News*, 1906, vol. 93, p. 271.)—It was asserted by Scheurer-Kestner that the solution of platinum in sulphuric acid was accelerated by the presence of nitrous products; but this was denied by Conroy (*Journ. Soc. Chem. Ind.*, xxii., 465), and Delépine (*Comptes Rendus*, 1905, 866, 1013) found that the addition of nitric acid had no influence on the speed of solution. The author's experiments have been made with commercial and pure platinum and sulphuric acids of 94 per cent. strength upwards, the heating being done both *in vacuo* and in an atmosphere of oxygen. In the case of commercial platinum and 94 per cent. acid, 0·001 gram per square decimetre of metal was dissolved in an hour *in vacuo*, whilst 0·124 gram was dissolved in oxygen. With sulphuric acid containing 2 per cent. excess of anhydride pure platinum was dissolved *in vacuo* to the extent of 0·0265 gram per square decimetre per hour, and sulphurous acid could be detected on opening the tube. The author concludes from these and other experiments that in the case of acid of the ordinary commercial strength it is atmospheric oxygen that acts as the oxidizing agent, whilst with acids of greater concentration the oxygen required is, in the absence of atmospheric oxygen, supplied by the sulphuric anhydride contained in solution in the sulphuric acid. C. A. M.

A New Reaction of Nickel. C. Reichard. (*Chem. Ztg.*, 1906, xxx., 556.)—If anhydrous nickel sulphate is intimately mixed with an equal quantity of methylamine hydrochloride ($CH_3NH_2.HCl$), and the mixture heated in a small porcelain crucible until white fumes are given off, a blue colour is developed, which is very delicate at first, but increases in intensity with the heating. On removing the source of heat, the colour is almost immediately changed back to that of the yellow nickel sulphate, but renewed heating will reproduce the blue colour as long as any methylamine hydrochloride is left. As little as 0·0001 gram of nickel will give a decided blue colour. The nitrate and chloride behave in the same way. If cobalt nitrate is heated, on the other hand, a blue colour is obtained whether methylamine hydrochloride is present or not, and this colour is not changed by interrupting the heating. The disappearing of the blue colour consequently appears to be characteristic of nickel. A. G. L.

A New Reaction of Tin. C. Reichard. (*Pharm. Centralh.*, 1906, vol. 47, p. 391; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 177.)—Any stannic compound—*e.g.*, stannic chloride—treated with caustic soda and then warmed with finely powdered uric acid in a porcelain crucible, yields a gray to intensely black stain. The reaction is not given by stannous compounds, arsenic, antimony, mercury, copper, lead, bismuth, or cadmium. A. G. L.

The Determination of Cadmium. C. Goldschmidt. (*Zeit. anal. Chem.*, 1906, xliv., 344.)—Cadmium is quantitatively precipitated when a solution of one of its salts is boiled in an aluminium vessel in the presence of a trace of chromium nitrate and cobalt nitrate. The precipitation is as complete as that of gold by nickel or of silver by cobalt, and is also successful in the presence of other metals. C. A. M.

Complete Analysis of Ferrochrome. G. Dillner. (*Jernkontorets Annaler*, 1906, vol. 60, p. 253; through *Chem Zeit.*, 1906, xxx., 178.)—Carbon is determined by combustion with lead peroxide in a current of oxygen. For chromium the sample is heated with sodium peroxide in a nickel crucible; the melt is taken up in boiling water, and chromium titrated with ferrous sulphate in the filtered and acidified solution. Iron is determined by titration in the insoluble residue from the melt, manganese being determined by Volhard's method in another similar residue. For silicon the sample is fused with sodium peroxide, and the melt obtained dissolved in hydrochloric acid; the solution is reduced with alcohol, chromium and iron hydroxides precipitated by ammonia, dissolved in hydrochloric acid, this solution evaporated to dryness, and silica determined as usual. In the filtrate phosphorus is determined. For sulphur another portion is fused with sodium peroxide. A. G. L.

The Detection and Determination of Traces of Iron. A. Mouneyrat. (*Comptes Rendus*, 1906, cxlii., 1049-1051.)—On treating 50 c.c. of the very dilute solution of the iron salt with 3 c.c. of ammonia solution (62 grams of NH_3 per litre) and passing a current of hydrogen sulphide for ten to twelve minutes, a dark green coloration is obtained, the iron apparently being present in a colloidal state. The test is very sensitive, and is capable of detecting 1 part of iron in 800,000. The green colour is destroyed by mineral acids and by many inorganic salts, such as sodium sulphate or chloride in concentrated solution. On the other hand, many organic substances increase the stability of the colloidal condition—*e.g.*, glycerin, glucose, mannite, as well as lactic, tartaric and citric acids, whilst albumin has a particularly marked effect in rendering the reaction more sensitive (1 in 1,000,000). Mercury, lead, silver, chromium, nickel, cobalt, copper, and metals of the alkaline earths, do not give any coloration. Copper, however, interferes with the test for iron, and should be removed by means of hydrogen sulphide. The intensity of the green coloration is proportional to the amount of iron for solutions containing 1 part in 1,000 to 1 in 1,000,000, and a colorimetric determination may be made with the aid of standard solutions of iron. C. A. M.

Modified Evolution Method for the Determination of Sulphur in Pig-Iron. J. McFarlane and A. W. Gregory. (*Chem. News*, 1906, vol. 93, p. 201.)—The powdered pig-iron is heated to redness with cream of tartar, and the hydrogen sulphide liberated by treating the mixture with hydrochloric acid titrated with standard iodine solution. Five grams of the powdered sample are intimately mixed with about 0·5 gram of cream of tartar, the mixture wrapped in filter-paper, placed in a small covered crucible, and heated to bright redness in a muffle for a quarter of an hour. After cooling, the resulting mass is powdered in a glass mortar and transferred to the evolution flask, where it is treated with boiling HCl (2 parts acid to 1 part water), the delivery tube from the flask dipping into an ammoniacal solution of cadmium chloride, prepared by dissolving 20 grams of the latter in a litre of water and adding a litre of strong ammonia. For each estimation 25 c.c. of this solution is diluted with about 300 c.c. of water, and placed in a tall beaker. The last traces of hydrogen sulphide are expelled from the flask by boiling, and the liquid containing the cadmium sulphide in suspension is acidified with hydrochloric acid, and the hydrogen sulphide in solution immediately titrated with standard iodine solution, each c.c. of which is equivalent to 0·00025 gram of sulphur, and standardized by means of a steel of known sulphur content. The cream of tartar should be free from sulphur. Results obtained by this process are found to agree very approximately with those found by the "aqua regia" method. W. H. S.

The Determination of Potassium in Potassium Salts and Artificial Manures. M. Kling and O. Engels. (*Zeit. anal. Chem.*, 1906, xlv., 315-332.)—The following simpler modification of the Finkener-Neubauer method is recommended: 10 grams of the potassium salt are dissolved in 500 c.c. of water, and 25 c.c. of the solution (= 0·5 gram) mixed with a few drops of hydrochloric acid and the required amount of platinum chloride, and evaporated to dryness in a porcelain basin. The residue is taken up with water and alcohol as in Neubauer's method, collected in a Neubauer-Gooch crucible, washed with alcohol, and dried by placing the crucible on a moderately hot metal plate. It is next reduced in a current of coal-gas, the heating being done with a very low flame for the first ten minutes, and finally at a faint red heat for twenty minutes. When the reduction is complete, the crucible is heated for two minutes over the naked flame of a Teclu burner, and, after its contents have been washed about fifteen times with hot water and two or three times with cold 15 per cent. nitric acid, it is placed in a porcelain vessel containing nitric acid, of the same strength, the level of liquid reaching to about three-quarters of the height of the crucible. The vessel is covered with a clock-glass, heated for thirty minutes on the boiling water-bath, and allowed to cool. Finally, the platinum in the crucible is washed with hot water and alcohol, ignited, and weighed, and the weight calculated into the corresponding amount of potassium. The accuracy of the method is shown by the results of test analyses of different potassium salts.

In the case of artificial manures, 20 grams of the sample are shaken with about 800 c.c. of water for thirty minutes in a litre flask, the liquid then made up to the mark and filtered, and the potassium determined in the filtrate. For the potassium 100 c.c. (= 2 grams) are treated with ammonia and ammonium carbonate in a 200 c.c. flask, the mixture made up to 200 c.c. and filtered, and 50 c.c. (= 0·5 gram) evaporated in a platinum basin. The residue is dried, and, after volatilization of the ammonium salts, ignited until it begins to fuse, and then taken up with hot water and a little hydrochloric acid. As a rule, solution is complete, but occasionally there is a residue, which must be filtered off. The liquid is treated with platinum chloride solution (2 c.c. of a 10 per cent. solution is usually sufficient), and evaporated to dryness, and the potassium determined as above described. It is shown that the presence of phosphates does not interfere with the accuracy of the results. The paper concludes with a table for calculating the amount of potassium (as K_2O) from the weight of platinum obtained from 0·5 gram of the substance. (Factor = 0·48108.) C. A. M.

Determination of Sulphur in Pyrites. C. R. Gyzander. (*Chem. News*, 1906, 93, pp. 213, 214.)—The sulphur is precipitated as $BaSO_4$ after reducing the iron to the ferrous state by hydroxylamine hydrochlorate. About 0·2 gram of the finely powdered ore is heated with a mixture of 5 c.c. concentrated HCl, and 15 c.c. concentrated HNO_3, evaporated to dryness, another 5 c.c. concentrated HCl added, and again evaporated. To the residue is added 100 c.c. distilled water, 1 c.c. concentrated HCl, and 3 c.c. hydroxylamine hydrochlorate solution, containing 1 ounce of the salt in 500 c.c. water, and when reduction is complete the solution is filtered, the filtrate heated almost to boiling, and 10 c.c. of a cold 10 per cent. $BaCl_2$ solution added. The $BaSO_4$ is then filtered off, washed, dried, and weighed. W. H. S.

Determination of Phosphorus and Ash in Coke. J. F. Hoy. (*Foundry*, 1906, vol. 28, p. 155; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 178.)—To determine ash, 1 gram of the finely-powdered coke is mixed to a thin paste with absolute alcohol in a platinum dish of 150 c.c. capacity; the dish is rotated slowly, so as to distribute the mixture uniformly over the whole of the inside, and the motion continued until the alcohol has evaporated. From five to seven minutes' heating over a blast-lamp or in a muffle is then sufficient to completely ash the coke. To determine phosphorus, the ash obtained as above is treated with 10 c.c. dilute hydrochloric acid and 29 (? 20) c.c. hydrofluoric acid, and the whole evaporated to dryness. The residue is taken up in 15 c.c. hydrochloric acid; the solution is transferred to a flask, and treated with ammonia, nitric acid, and molybdic acid, the determination then being finished as usual. A. G. L.

Chemical Methods for the Examination of Coal Briquettes and Briquette Pitch. E. J. Constam and R. Rougeot. (*Glückauf*, 1906, vol. 42, p. 481; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 178.)—Briquettes should be made from hard coal, but not anthracite, and should contain a certain amount of binding material. The coal used, and the briquettes themselves, should be examined for coke yielded on carbonizing. In Germany, Austria, and Switzerland, Muck's method is used for this purpose. Broockmann's "Bochum" test gives results 1 to 3 per cent. lower, but in good accord with the results obtained in gasworks. The Belgian method gives variable results. The American method gives the best results. It consists in heating 1 gram of the undried coal in a covered platinum crucible weighing 20 to 30 grams in a Bunsen flame 20 cm. high, at a point 6 to 8 cm. above the burner. To determine the coke yielded by the pitch used in making briquettes, the

Bochum method is to be preferred. The amount should not exceed 45 per cent. The softening and melting points of the pitch should be noted, and also its behaviour in the molten state. The amount soluble in carbon bisulphide varies from 60·4 to 95 per cent., the average being 79·8 per cent. The calorific value of the pitch should not be below 8,550, and the quantity of hydrogen present should not exceed 5 per cent.

To determine the amount of binding material in the briquettes, the sample should be twice extracted with carbon bisulphide in a Soxhlet for twenty-four hours at a time, the extract being dried for three days over phosphorous pentoxide *in vacuo*. The amount obtained should not be less than 5 per cent. The ultimate composition of the briquettes should also be determined. The calorific value of the dry briquette should not be less than 7,700, the ash not more than 8 per cent., and the amount of volatile matter not less than 16 per cent.
A. G. L.

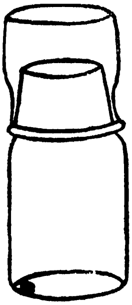
On the Determination of Water-soluble and Total Phosphoric Acid in Superphosphate. K. Rohm. (*Chem. Zeit.*, 1906, xxx., 542.)—*Water-soluble Phosphoric Acid.*—A number of determinations were made on three samples to compare the method in which the sample is digested with water for two hours, with occasional shaking, with that in which it is agitated continuously with water for thirty minutes in a shaking machine, different speeds being also tested. The most constant results are given by the latter method, the speed being about thirty to forty rotations per minute, but digesting the sample without any shaking at all gave results only about 0·2 per cent. too low (with 19 per cent. of water-soluble P_2O_5). The sample used should pass through a 2 mm. mesh sieve.

Total Phosphoric Acid.—Extraction with nitric acid, with a mixture of nitric and sulphuric acids, and with aqua regia, gave practically identical results. Extraction with hydrochloric acid, which is never used in practice, gave results about 0·2 per cent. too low. The magnesia precipitate should be allowed to stand twenty-four hours before filtering, or else mechanically stirred for thirty minutes. On allowing it to stand for only two hours, an error of up to 0·22 per cent. of P_2O_5 was found in a number of analyses.
A. G. L.

On the Determination of Nitric Acid in Water. Paul Drawe. (*Chem. Zeit.*, 1906, xxx., 530.)—The author has altered Frerich's method (*Arch. d. Pharm.*, 1903, 241, 47) as follows: If nitrates have been qualitatively shown to be present, 100 c.c. of the water are evaporated to dryness with an excess of hydrochloric acid. The residue is evaporated several times with water, until every trace of free acid is removed, after which the chlorine in the residue is determined by titration. From the value found the chlorine present in the water itself, plus that due to the alkalinity of the original water, is deducted, and the remainder calculated to nitric acid. The method is said to give good and accurate results.
A. G. L.

APPARATUS.

A New Weighing-Bottle.—This improved form has many decided advantages over those in use, being easier and more cleanly to work with, and insures greater accuracy in weighing. Also, it may be used for purposes for which the old form was not adapted. The old form has the stopper ground to fit inside the neck; the new one has the stopper ground to fit like a cap over the neck of the bottle. The advantages claimed are: (1) On tipping out many substances from the old form of bottle, some remains sticking to its lip, and is left exposed to the air when the stopper is replaced, thus absorbing moisture. With the improved stopper the lip remains enclosed, and any substance adhering to it cannot absorb moisture from the air, and so affect the weighing. (2) With the old form, on replacing the stopper after tipping out any substance, it is usually found that some substance remains attached to the inside of the neck, thereby getting ground in between the stopper and the neck, causing the stopper to fit inaccurately, and, if left for some time, this makes the stopper stick so firmly that it is generally broken on trying to remove it. The stopper being outside obviates all this. (3) The stopper, when placed on the bench or table, does not roll about, but lies flat. The old form of stopper rolls about, and is likely to have some substance sticking to it, some of which falling off on to the bench, is lost, and affects the weighing. The new weighing-bottle is generally much cleaner to work with. (4) Liquids are more easily dealt with. The bottle may be obtained of Messrs. J. Kerr and Co., Manchester. W. J. S.



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PRESERVATIVES IN MILK.

The following Circular has been addressed to the various Local Authorities under the Sale of Food and Drugs Acts:

Local Government Board,
Whitehall, S.W.
July 11, 1906.

Sir,—I am directed by the Local Government Board to request the attention of the Council to the subject of the addition of preservatives to milk.

A serious objection to the use of preservatives in milk has been pointed out in the report of the Departmental Committee on Preservatives and Colouring Matters in Food, who state that preservatives in milk "may be relied on to protect those engaged" in the milk traffic "against the immediate results of neglect of scrupulous cleanliness. Under the influence of these preservatives milk may be exposed without sensible injury to conditions which otherwise would render it unsaleable. It may remain sweet to taste and smell, and yet have incorporated disease germs of various kinds, whereof the activity may be suspended for a time by the action of the preservative, but may be resumed before the milk is digested."

This Committee, after hearing evidence from milk traders, concluded that the addition of a preservative to milk is not necessary for the purposes of the milk trade, even in hot weather or where the supply of so large a place as London is concerned, and the Committee recommended that no preservatives should be added to milk.

In making this recommendation the Committee had special regard to evidence received as to two classes of preservative substances which, under various names, are frequently used as preservatives in milk—viz. : (1) Formalin (a 40 per cent. solution of formic aldehyde) and other preparations of formic aldehyde ; and (2) boron preservatives (boric acid, borax, or mixtures of boric acid and borax). The Committee considered that the addition to milk of formalin or preparations of formalin, even when the amount which could be detected was minute, was objectionable, on account of the alterations effected by formalin in the character of certain of the constituents of milk and of its ability to interfere directly with digestive processes.

Although in the view of the Committee boron preservatives might reasonably be employed in the case of certain foods, within defined limits and subject to a declaration as to their presence and amount, the Committee recommended their exclusion from milk altogether; partly for the reasons above indicated, and partly also in consideration of the immense importance of pure milk for the nutrition of infants, invalids, and convalescents, and of the comparatively large quantity of milk which may be taken, particularly by children, in comparison with the other foods in question. Moreover, the Committee had evidence "pointing to an injurious effect of boracized milk upon the health of very young children."

Since the report of the Committee was made, the Board have from time to time had before them further evidence on the subject, and this supports the conclusions of the Committee not only as to the objections to the use of preservatives on the ground of public health, but also as to the ability of milk traders to conduct their business without use of preservatives. Thus in certain boroughs in London and elsewhere in which milk samples are systematically tested for preservatives, the presence of preservatives in milk at any time of the year has been found to be exceptional ; and there is evidence to show that a very large number of milk vendors conduct their business without the use of these substances, even where the milk comes long distances by rail.

In some districts action under the Sale of Food and Drugs Acts has been frequently and successfully taken in order to bring about the disuse of preservatives in milk. Proceedings instituted against vendors of milk containing preservatives have usually been taken under Section 6 of the Sale of Food and Drugs Act, 1875. Conviction has followed, it being held that when the purchaser who asks for milk is supplied with milk plus a preservative, he does not receive an article of the nature, substance, and quality demanded, and is prejudiced thereby.

The Board are of opinion that action under the Sale of Food and Drugs Acts in regard to preservatives in milk is desirable, and that this subject deserves attention from all authorities in England and Wales charged with the execution of these Acts.

In this connection the following suggestions are made for adoption by the Council where a similar procedure is not already followed:

1. *Information from Public Analysts.*

The Board suggest that Public Analysts should be requested—

(*a*) To record in their quarterly reports how many milk samples have been examined during the quarter with a view to ascertaining the presence of substances commonly in use as preservatives, and with what result ; and to draw the attention of the Council to instances where the use of preservatives in milk other than boron preservatives and formalin have come under notice.

(*b*) To report, on completion of analysis, the facts as to samples of milk which have been found to contain any added preservative.

2. *Administrative Action where Preservatives in Milk are reported.*

The Board would suggest that the Council should notify to milk traders, by circular or otherwise, that action will be taken under the Sale of Food and Drugs Acts in instances where preservatives are reported in milk.

Subject to this being done, and to exceptional cases of the kind referred to under the heading numbered 3 below, the Board consider that when the presence of any added preservative is reported in a sample of milk taken in accordance with the provisions of the Sale of Food and Drugs Acts, the case should in ordinary circumstances be regarded as one for the institution of proceedings under those Acts.

3. *Declaration and Notices.*

The Board think it desirable to draw attention to cases in which the vendor of the milk, with the object of escaping liability under Section 6 of the Sale of Food and Drugs Act, 1875, declares to the purchaser by means of a notice, label, or otherwise, that he does not sell " milk " as such, or that its quality in regard to preservatives or other constituents is not guaranteed, or that it contains some added preservative.

The Board would suggest the desirability of frequent sampling in cases where " milk " is sold subject to declarations of the kind, with a view to ascertaining the condition of such milk in regard to preservatives.

The nature of the declaration made should in all cases be carefully recorded by the officer taking the sample, and should also be reported to the analyst when the sample is transmitted for analysis.

Where preservatives are reported in milk thus sold, the question will arise whether, in view of the nature and quantity of the preservatives added, it can be considered that the article has been rendered injurious to health, or that the purchaser has been prejudiced, to an extent which would justify the institution of proceedings under Section 3 or Section 6 of the Sale of Food and Drugs Act, 1875, notwithstanding the declaration made at the time of purchase.

This question is not without difficulty in view of the general objection to the employment of any preservatives in milk referred to above.

As regards formalin and boron preservatives, however, the Board are advised that the presence in milk of formalin to an amount which is ascertained by examination *within three days of collecting the sample* to exceed 1 part in 40,000 (1 part in 100,000 of formic aldehyde) raises a strong presumption that the article has been rendered injurious to health, and that the purchaser has been prejudiced in the above sense ; and also that similar presumption is raised where boron preservatives are present in milk to an amount exceeding 40 grains of boric acid per gallon.

It appears desirable that the addition of preservatives to skim milk, separated milk, and condensed milk should be watched and controlled on similar lines.

Additional copies of this Circular are enclosed for transmission to the Public Analyst, and for use by executive officers under the Sale of Food and Drugs Acts. The Circular will be placed on sale, and copies can then be obtained from Messrs. Wyman and Sons, Limited, 109, Fetter Lane, Fleet Street, London, E.C., either directly or through any bookseller.

I am, Sir,
Your obedient servant,
S. B. Provis,
Secretary.

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REVIEWS.

Studies on Immunity. By Professor Paul Ehrlich and his Collaborators. Translated by Dr. Charles Bolduan. (New York: John Wiley and Sons. London: Chapman and Hall, 1906. Pp. 586. Price 25s. 6d. net.)

Ehrlich's studies on immunity have acquired a world-wide reputation, and all investigators in this important field of study will welcome an English translation of the work of himself and his collaborators. In his preface the author points out that Behring's great discovery of antitoxin opens new paths for research, first in the direction of the production of individual curative sera, and, secondly, in seeking a deeper insight into the nature of immunity phenomena.

Ehrlich's theories are developed on the basis of chemical conceptions, and he shows that the significance of morphological structure is far less than that of the chemistry involved. By a comparison with the mechanical conditions and suitable apparatus that aid, but are not absolutely necessary to a chemical process that depends essentially on the constituents involved, so in biology the morphological arrangement of the organs and cells is not the essential feature. This is rather to be sought for in chemical differences of the constituents.

The products of bacteria, as well as the artificially-produced bacteriolysins and most of the ferments, according to the author, produce their effects by the presence of two active groups in the molecule, one of these uniting with the substance to be acted upon, whilst the other produces the characteristic effect. This theory is bound to affect the study of vital phenomena, especially intracellular metabolism and other physiological problems, such as those of secretion, heredity, etc.

The book comprises forty-one chapters by various investigators on the subject of immunity, including many by Ehrlich himself.

One of these is of special interest from the chemical standpoint, consisting as it does of a discussion of the relations existing between chemical constitution, distribution, and pharmacological action. The author here shows that at the present time the chemical aspect constitutes the axis about which the most important views in medicine turn, and that the two poles are the synthetic construction of new therapeutic agents on the one hand, and the discovery of specific therapeutic products of living cells on the other.

He also enumerates the conclusions that can be drawn from the study of the large number of medicaments of therapeutic importance. In this synopsis the fact is emphasized that the antipyretic power of antipyretics is destroyed by the introduction of salt-forming acid radicals, such as SO_3H and $COOH$, and cites as an illustration of this acetanilido-acetic acid ($C_6H_5N(COCH_3)CH_2COOH$), and acetanilin sulphonic acid ($C_6H_5NH.CO.CH_2.SO_3H$), which are inert in this respect.

Ehrlich also mentions that the presence of the ethyl group in certain disulphons—for example, sulphonal [$(CH_3)_2C(SO_2C_2H_5)_2$] and trional [$CH_3C_2H_5C\ (SO_2C_2H_5)_2$], as shown by Baumann—gives to these substances their hypnotic property, and that the latter increases with the number of these groups.

Another interesting chapter is that by Dr. Preston Kyes and Dr. Hans Sachs on the substances which activate cobra venom.

The book is well arranged, and should prove of great interest and value to all students and workers in the important subjects of immunity and vital phenomena.

Official Chemical Appointments. By R. B. Pilcher. (London: Institute of Chemistry. Price 2s. net; post free, 2s. 3d.)

Whilst the clerical, legal, and medical professions have been long since well supplied with copious directories, giving, amongst other things, lists of appointments held by members of each respective profession, no approach to such convenient works of reference has been hitherto compiled for the chemical profession. In order to fill up this void, Mr. R. B. Pilcher, the able Registrar of the Institute of Chemistry, has, under the direction of the Council of that body and under the supervision of its Proceedings Committee, undertaken the compilation of such a work. The list of official appointments is in two divisions, the former embracing the holders of appointments in Great Britain and Ireland, the latter of those in our colonies; that of appointments held by Public Analysts, which was published some months since, has also been incorporated with the work. In order to be as comprehensive as possible, appointments in connection with agriculture, metallurgy, assaying, and other branches of work where chemical knowledge is required are mentioned. In many instances particulars are given as to the Acts under which appointments are made, as well as the regulations and conditions of such appointments. The work also contains a large quantity of useful miscellaneous information on many subjects.

Mr. Pilcher is to be congratulated on the excellent manner in which he has performed his arduous task; he has succeeded in producing a very useful and highly interesting book for members of the profession to whom it relates, as well as to many outside that profession. W. J. S.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

Pass List of the July Examinations.

Of fourteen candidates who entered for the Intermediate Examination, the following nine passed: L. C. W. Bonacina, W. R. S. Ladell, D. J. Law, W. M. Seaber, B.Sc. (Lond.), P. Stutfield, J. M. Weir, M.A., B.Sc. (St. Andrews), W. A. Whatmough, J. M. Wilkie, B.Sc. (Lond.), and C. H. Wright, B.A. (Cantab.). In the Final Examination for the Associateship (A.I.C.), of three examined in the branch of Mineral Chemistry, two passed: J. W. Agnew and I. M. Heilbron. Of three in the branch of Organic Chemistry, two passed: R. Le Rossignol, B.Sc. (Lond.), and G. W. Monier-Williams, M.A. (Oxon.), Ph.D. (Freiburg). And of eight who entered in the branch of the Analysis of Food and Drugs and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following six passed: J. G. Annan, B.Sc. (Lond.), C. T. Bennett, B.Sc. (Lond.), G. W. Glen, F. W. Harris, E. H. Merritt, B.Sc. (Lond.), and F. Tattersfield. The Examiners in Chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A. (Glasgow), F.I.C. The Examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A. (Cantab.), F.R.S., F.I.C.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON THE EXAMINATION OF OLIVE, LINSEED, AND OTHER OILS.

By R. T. THOMSON and H. DUNLOP.

(*Read at the Meeting, June* 14, 1906.)

Having seen reason, after careful investigation, to adopt Wijs' method of determining the iodine value of oils in place of the Hübl method, we have made an examination of various oils of undoubted genuineness, and propose in the present paper to describe the results. As we have stated before, there is an element of uncertainty and unreliability in the Hübl method, and in any event the Wijs and Hübl results cannot be depended on as being interchangeable. It is therefore of importance that a fresh series of iodine values by the Wijs method should be constructed, and this we have done for several oils in the present paper, and embodied the results, along with those of other constants, in Tables I. and II., p. 282.

In the case of the olive oils prepared from the olives by ourselves, those extracted by carbon bisulphide were from the residue left after pressing out the bulk of the oil, and it will be observed that there is little difference between the two varieties. It is evident from these results that a genuine olive oil may vary in iodine value from 81 to 89, and may be regarded as such if the other constants are normal. There is a peculiarity with regard to the Mogador oil, which has an extremely high iodine value, while the refractometer reading is lower than would be expected. We have found with regard to linseed and fish-liver oils that the iodine value and refractometer reading practically rise and fall simultaneously, but olive oil appears to be quite erratic in this respect, as will be seen by an inspection of the tables. This difference is no doubt partly due to the influence of the free fatty acids, which, it has been stated, lower the refractive power; and this is undoubtedly the case, as will be seen from Table III., p. 283.

TABLE I.

Oils extracted by ourselves from the Seed or Fruit, so that their Genuineness can be absolutely depended upon.

	Iodine Value (Wijs).	Zeiss Refractometer at 25° C.	Saponification Value. Per Cent.	Unsaponifiable Matter. Per Cent.	Specific Gravity, 60° F.	Free Acid Per Cent.
Olive oil (Spanish, green, by pressure)	83·20	61·2	19·56	1·25		1·28
Olive oil (Spanish, green, by CS_2)	83·20	61·2	19·21	1·62		0·90
Olive oil (Spanish, ripe, by pressure)	88·95	61·3	19·28	1·34	0·9156	0·51
Olive oil (Spanish, ripe, by CS_2)	88·15	62·2	19·14	1·52		0·36
Olive oil (Turkish, very ripe)	89·1	61·2	19·21	1·24	0·9156	9·37
Linseed oil (Riga seed)	205·4	85·5	19·21	1·25	—	
Linseed oil (St. Petersburg)	200·0	84·2	19·28	1·23		
Linseed oil (North American)	194·6	83·2	19·21	1·10		
Linseed oil (Calcutta)	188·6	81·7	19·28	0·88		
Linseed oil (River Plate)	185·5	81·0	19·14	1·25		
Ravison oil	118·1	71·0	18·13	1·65		
Jamba oil	98·3	67·2	17·53	1·02		
Rape oil (East India) ...	104·5	68·0	17·53	1·02		
Almond oil	98·1	64·3				
Castor oil	85·6	78·3	18·16	0·60		

TABLE II.

Oils obtained from Reliable Sources.

	Iodine Value (Wijs).	Zeiss Refractometer at 25° C.	Saponification Value. Per Cent.	Unsaponifiable Matter. Per Cent.	Specific Gravity, 60° F.	Free Acid. Per Cent.
Olive oil (Crete)	81·2	60·2	19·14	—	0·9155	9·40
,, (Italian) ...	83·5	59·7	19·21	—	0·9157	16·61
,, (Sicilian) ...	84·1	60·0	19·07	—	0·9144	11·50
,, (Levant) ...	84·4	61·0	19·21	—	0·9159	9·32
,, (Algerian) ...	85·1	60·7	19·14	—	0·9150	5·62
,, (Syrian) ...	85·3	60·1	19·14	—	0·9145	11·76
,, (Spanish) ...	86·6	61·2	19·21	—	0·9161	7·27
,, (Mogador) ...	94·3	60·5	19·07	—	0·9150	24·72
Poppy-seed oil	140·0	71·0	19·28	0·52	0·9243	1·62
Sunflower oil (Russian) (by Hübl's method) ...	131·3	70·0	18·93	0·70	0·9220	1·21
Arachis oil	87·5	62·6	19·14	—	0·9164	—

TABLE III.

Showing the Effect of Free Fatty Acids on the Refractive Power of Olive Oil.

	Olive Oil (Mogador).	Olive Oil (Italian).
Iodine value before removal of free acid	94·30	83·50
Iodine value after removal of free acid	93·45	80·45
Zeiss refractometer, 25° C. before removal of free acid	60·50	59·70
Zeiss refractometer, 25° C. after removal of free acid	63·40	61·00
Free oleic acid, per cent. before removal of free acid	24·72	16·61
Free oleic acid, per cent. after removal of free acid	0·32	0·27

TABLE IV.

Comparison of Iodine Value and Refractive Power of Linseed and certain Fish-liver Oils.

	Iodine Value.	Zeiss Refractometer at 25° C.
Linseed oil (Riga)	205·4	85·5
,, (St. Petersburg)	200·0	84·2
,, (North America)	194·6	83·2
Skate-liver oil	191·1	82·5
Linseed oil (Calcutta)	188·6	81·7
Haddock-liver oil...	186·4	81·0
Linseed oil (River Plate)...	185·5	81·0
Whiting-liver oil	184·2	81·0

The differences referred to cannot, however, be entirely explained by the influence of the free acid, as will be apparent from an examination of the results in Tables I. and II., and especially of those of the two oils extracted from the same Spanish ripe olives, where the oil with the lower iodine value shows the higher refractive power, the free acids being practically the same.

The different samples of linseed from which we prepared the oils were carefully examined, and any foreign seeds removed, so that only the genuine linseed was employed. It will be observed that the iodine value for the oil extracted from the Riga seed is, we believe, the highest on record, while that from the River Plate has an iodine value much higher than what was recorded by one of us some years since. There can be no doubt, however, that these high figures are due partly to the use of the Wijs in place of the Hübl method, and where the former is employed any iodine value below 180 should lead to a more searching examination of the oil.

As regards the other oils of which the constants are included in the tables, it is scarcely necessary to make any remarks, except that the jamba oil cannot be distinguished from rape oil by ordinary means, and that in some few cases the employment of the refractometer along with the iodine value may be useful, although the former is of no independent importance.

In conclusion, we would draw particular attention to the peculiar similarity between various linseed oils and certain fish-liver oils as regards iodine value and refractive power, as shown in Table IV., p. 283.

Of course, linseed oils have a distinctly higher specific gravity than the fish-liver oils, but the saponification value and unsaponifiable matter are much the same in each.

* * * * *

ON THE COMPOSITION AND VALUATION OF OILS USED FOR GAS-MAKING PURPOSES.

By Raymond Ross, F.I.C., and J. P. Leather.

(Read at the Meeting, June 14, 1906.)

A large number of investigations have been made by numerous workers into the composition of the various petroleum oils found in certain parts of the world. These investigations have been largely confined to the fractions of low boiling-point, and their principal aim has been the separation and isolation of definite compounds.

The aim of our work has been primarily directed to the valuation of those oils which are used for gas-making purposes. Such oils consist generally of those portions of the crude oil which boil between 200° C. and 400° C. In certain states of the oil market even the refined petroleum burning oils are also used.

From the results obtained by previous investigators—to which we shall refer later—we know that the various classes of hydrocarbons are found in varying proportions, according to the origin of the oil.

This being the case, it is evident that to obtain a satisfactory knowledge of the gas-making properties of any oil, it is necessary to ascertain the proportion of the different classes of hydrocarbons present in the oil, and the gas-making properties of each class. The presence of the following classes of compounds in given petroleum oils has been proved by various workers—notably, Markownikoff, Zelinsky, Aschan, Schorlemmer, Engler, Schutzenberger, Maybery, and others:

Paraffins.
Olefines.
Aromatic hydrocarbons.
Naphthenes.

In this last term are included saturated and unsaturated hydrogenated derivatives of both benzene and naphthalene and their homologues. Some preliminary work which we undertook—the result of which was communicated to the Society of Chemical Industry (May, 1902)—led us to think that the determination of certain chemical and physical properties of an oil might enable a correct judgment to be formed of the nature and composition of any gas oil. For this purpose it was necessary that the physical properties and the value for gas-making properties of each class of hydrocarbons should be separately determined. The present communication sets forth the progress that we have so far made in this direction, and also the results obtained from the examination of a large number of gas oils.

Although, owing to the difficulties inherent to such a research, our progress is more limited than we had hoped, we trust that our results will be found to be both of theoretical interest and practical utility.

Before proceeding further it will perhaps be best to recapitulate the methods which we use for the valuation of an oil, and which are substantially those mentioned in our previous paper.

For the purpose of gasifying the oil we make use of a retort about 9 inches long by 5¾ inches wide and 4¾ inches in height. This is heated in a muffle furnace. The retort carries an electrical pyrometer and two tubes—one for introducing the oil, going only a short distance into the retort, and the other for the exit of the gas, being carried the whole length of the retort.

The oil is fed in from a burette, and the gas, after passing through a tar-bottle, is collected in a holder, measured, and analysed. After the retort has been brought to the required temperature, the gas-supply is turned off, and 15 c.c. of oil run in during a period of about three minutes.

Experience has shown us that if these directions are followed there is practically no variation in the temperature of the retort during the period of gasification. The size of the retort as well as the rate of flow of the oil are important factors, as if the time of contact is altered, the quantity and quality of the resultant gas are affected. The resultant gas is measured, and then the amount of hydrocarbons absorbed by fuming sulphuric acid is ascertained in a Hempel burette, and this figure is given in all our results as "hydrocarbons."

The number of c.c. of gas at N.P.T. per c.c. of oil (at 15° C.), multiplied by the percentage of hydrocarbons ascertained as above, gives a number which we have called the "valuation figure." The temperatures which we have selected for cracking the oils are those at or between which we have, after many experiments, invariably obtained the highest valuation figure.

In addition to the foregoing cracking test, we make a distillation test on the following lines: 250 c.c. of the oil are distilled, and the fractions are separated at intervals of 20° C. in such a way as to have an even number for the second digit. The quantity of each fraction is measured, and its specific gravity and refractive index determined. The specific refraction is then calculated. Before proceeding further it will, perhaps, be best to give a résumé of the results which we have obtained by the examination of a number of different oils from various parts of the world.

DISTILLATION TEST.

	200° C. to 220° C.			220° C. to 240° C.			240° C. to 260° C.			260° C. to 280° C.			280° C. to 300° C.			300° C. to 320° C.			320° C. to 340° C.			340° C. to 360° C.			360° C. to 380° C.			380° C. to 400° C.		
	Gravity at 15° C.	N_D 20° C.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$	Sp. Gr.	N_D.	$\frac{N-1}{d}$
Pennsylvanian:																														
	—	—	—	—	—	—	0·797	1·4449	0·558	0·821	1·4570	0·556	0·833	1·4635	0·556	0·841	1·4681	0·556	0·851	1·4734	0·555	0·862	1·4789	0·555	0·871	1·4844	0·555	0·877	1·4879	0·556
I.	—	—	—	—	—	—	—	—	—	0·002	0·0014	0·001	—	0·0004	0·001	—	0·0003	0·001	0·001	0·0007	0·001	0·001	0·0005	—	0·001	0·0004	0·001	0·001	0·0004	—
Kansas:																														
	0·804	1·4475	0·556	0·816	1·4534	0·555	0·829	1·4597	0·555	0·838	1·4655	0·555	0·850	1·4723	0·555	0·860	1·4780	0·556	0·867	1·4816	0·555	0·874	1·4866	0·557	0·882	1·4922	0·558	—	—	—
Russian Refined:																														
	0·825	1·4547	0·551	0·835	1·4605	0·551	0·844	1·4659	0·552	0·853	1·4711	0·552	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Russian:																														
	—	—	—	—	—	—	0·852	1·4679	0·549	0·855	1·4705	0·551	0·862	1·4749	0·551	0·871	1·4794	0·550	0·879	1·4829	0·549	0·887	1·4866	0·551	0·897	1·4929	0·549	—	—	—
Texas:																														
	0·844	1·4607	0·546	0·858	1·4701	0·547	0·873	1·4775	0·548	0·884	1·4849	0·548	0·895	1·4918	0·549	0·906	1·4974	0·549	0·915	1·5042	0·551	0·923	1·5093	0·551	0·928	1·5131	0·552	—	—	—
I.	—	0·0005	0·001	0·012	0·0044	0·003	0·014	0·0028	0·004	0·003	0·0019	0·004	0·003	0·0022	0·003	0·004	0·0033	0·005	0·003	0·0028	0·002	0·003	0·0029	0·001	0·003	0·0036	0·002	—	—	—
Californian:																														
	0·822	1·4554	0·554	0·836	1·4628	0·553	0·851	1·4709	0·553	0·864	1·4791	0·554	0·880	1·4873	0·554	0·895	1·4989	0·557	0·908	1·5040	0·555	0·920	1·5115	0·556	0·930	1·5197	0·559	0·989	1·5282	0·562
Roumanian:																														
	0·829	1·4616	0·556	0·847	1·4709	0·556	0·861	1·4796	0·556	0·873	1·4864	0·557	0·882	1·4914	0·557	0·883	1·4920	0·557	0·890	1·4959	0·557	0·894	1·4982	0·557	0·901	1·5021	0·557	—	—	—
I.	0·015	0·0080	0·004	0·009	0·0061	0·002	0·011	0·0064	0·002	0·012	0·0073	0·002	0·012	0·0057	0·002	0·017	0·0097	0·002	0·018	0·0099	0·002	0·014	0·0060	0·002	0·008	0·0028	0·002	—	—	—
Galician:																														
	—	—	—	—	—	—	—	—	—	—	—	—	0·856	1·4722	0·557	0·865	1·4830	0·558	0·876	1·4895	0·559	0·888	1·4961	0·559	0·904	1·5066	0·560	0·916	1·5134	0·560
Grosny:																														
	—	—	—	—	—	—	0·857	1·4719	0·550	0·860	1·4750	0·552	0·878	1·4847	0·553	0·886	1·4886	0·552	0·894	1·4947	0·554	0·902	1·4993	0·553	0·908	1·5044	0·555	0·917	1·5071	0·553
I.	—	—	—	—	—	—	0·006	0·0014	0·002	0·018	0·0084	0·002	0·007	0·0013	0·003	0·007	0·0028	0·001	0·007	0·0015	0·003	0·006	0·0029	0·001	0·005	0·0047	0·004	0·001	0·0022	0·003
Borneo:																														
	0·861	1·4827	0·560	0·879	1·4915	0·560	0·900	1·5048	0·560	0·920	1·5161	0·561	0·936	1·5242	0·560	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Scotch:																														
	—	—	—	—	—	—	—	—	—	0·833	1·4673	0·560	0·844	1·4732	0·560	0·853	1·4779	0·560	0·864	1·4851	0·561	0·877	1·4920	0·561	0·891	1·5012	0·562	0·904	1·5095	0·563
I.	—	—	—	—	—	—	—	—	—	0·007	0·0035	0·001	0·004	0·0023	0·001	0·004	0·0027	0·001	0·002	0·0023	0·001	0·009	0·0060	0·001	0·001	0·0011	0·000	—	—	—

N_D = Refractive index for Sodium light. $\frac{N-1}{d}$ = specific refraction.

Lines marked I. give the extreme variation from the average.

AVERAGES OF GASIFICATION RESULTS.

Name of Oil.	Temperature of Cracking.	C.c. of Gas N.T.P. per c.c. of Oil.	Hydrocarbons per Cent.	Valuation Figure.
Pennsylvanian ...	1260° F.	445	35·8	15,944
	1400° F.	529·9	30·1	15,950
	1510° F.	563	26·6	14,967
Kansas	1260° F.	438·9	33·6	14,747
	1400° F.	482·6	28·4	13,706
Russian	1260° F.	465·7	34·2	15,927
	1510° F.	556	22·8	12,677
Russian refined ...	1260° F.	429	31·8	13,642
	1510° F.	518	28·4	14,711
	1700° F.	550	21·5	11,825
Texas	1130° F.	325	30·1	9,769
	1260° F.	388·3	29·8	11,490
	1400° F.	461·8	25·3	11,697
	1450° F.	547·7	18·6	10,187
	1510° F.	508·8	21·1	10,741
Californian ...	1130° F.	370·8	29·8	11,050
	1260° F.	529·9	26·6	14,095
	1400° F.	573·9	25·4	14,577
Roumanian ...	1130° F.	301·3	40·5	12,302
	1260° F.	388·8	33·3	12,895
	1400° F.	459·7	28·6	13,147
	1450° F.	557·3	20·2	11,257
Galician	1260° F.	452·8	35·5	16,074
Grosny	1130° F.	341·8	34·9	11,925
	1260° F.	421·9	34·6	14,489
	1400° F.	501·6	25·8	12,860
Borneo	1260° F.	301	26·8	8,067
	1510° F.	472	17	8,024
	1640° F.	495	15	7,425
Scotch	1130° F.	286·5	40·3	11,548
	1260° F.	426·4	34·2	14,620
	1450° F.	491·5	26·1	12,790

As the worst results which we have obtained were got from Borneo oil, we determined to make a special examination of this oil.

We commenced work on the lower boiling fractions with an attempt to obtain some definite compound by means of repeated fractional distillations, but without success. We therefore had recourse to chemical means, and to this end treated that fraction of the oil which boiled in the neighbourhood of 200° C. with an excess of fuming nitric acid for a period of some days with constant mechanical stirring, adding the nitric acid gradually, and warming when the violence of the reaction had abated. At the end of the time the mixture separated into three layers—viz., unacted on oil, a wax-like substance, and nitric acid. The residual oil was treated with sulphuric acid and then heated with fuming sulphuric acid (50 per cent. SO_3) severa times. It has been shown that fuming nitric acid acts in the following manner (Francis and James, *Chem. Soc. Journ.*, 1898)—viz., it readily attacks aromatic compounds, methyl derivatives of polymethylene compounds, and the iso-paraffins. All these compounds are characterized by the presence of a CH group. Consequently in the fraction experimented upon only normal paraffins and decahydronaphthalene could be present in any quantity after the double treatment. An analysis of the remaining oil gave the following figures :

	Per Cent.	
C	86·27	Specific gravity of oil, 0·821 15°/15°
H	14·13	

This analysis shows less hydrogen than corresponds to C_nH_{2n}, and it therefore approximates more nearly to decahydronaphthalene than to a paraffin.

The oil was next boiled with excess of bromine in carbon tetrachloride with a reflux condenser for many hours, in the hope that some substitution might take place in the paraffins. Hydrobromic acid was given off, and after distilling off the carbon tetrachloride, the residue was boiled with caustic soda to decompose the bromine compounds and thus leave unsaturated products. After washing, the oil was again treated with fuming sulphuric acid and warmed. After separation, washing, and drying, the oil gave on analysis :

		Theory for $C_{10}H_{18}$.
C	86·77	86·96
H	13·24	13·04

Its specific gravity was 0·843 at 15° C. The N_D 1·4507. Boiling-point, 169·5° C. Molecular weight determination by Beckmann's boiling - point method, 133·4 (theory 138). According to Beilstein the gravity is 0·847 at 15° C., and the boiling-point 173° to 180° C. The substance obtained, which was probably fairly pure decahydronaphthalene, was then cracked in a similar manner to the gas oils at 1260° F., and gave results as below :

434·1 c.c. of gas at N.T.P. per c.c. of oil containing 26·2 per cent. of hydrocarbons soluble in fuming sulphuric acid, giving a valuation figure of 11,373.

The yellow wax-like substance obtained by nitrification was washed, separated, and dissolved in absolute alcohol. After many attempts two batches of crystals were obtained, in which the carbon was estimated by moist combustion with chromic acid. Several ordinary combustions were spoilt by explosions, even when air was used and great care exercised. The gas from the moist combustion was led through a combustion-tube containing copper oxide and reduced copper. The first batch of crystals gave, as a result of three closely agreeing analyses, carbon 54·28 per cent., and the nitrogen, as determined by the modified Kjeldahl method, was 12·7 per cent. These figures point to a dinitrotetrahydronaphthalene, for which the theory is C = 54·04 per cent. and N = 12·61 per cent. To verify this suggestion we treated about 70 grams of the nitro compound with 300 grams of tin and 1 litre of hydrochloric acid, heated under a reflux condenser until the tin was dissolved; we then added an excess of strong caustic soda. A thick, pitchy mass rose to the top. This was washed with water and finally distilled *in vacuo*.

To the distillate hydrochloric acid was added, and after standing crystals were obtained, which, on being recrystallized from alcohol, were nearly colourless, and gave on analysis—

	Per Cent.			Theory for $C_{10}H_{10}(NH_2HCl)_2$.
C	50·7	... ...	C	51·1
H	7·4	... ...	H	6·8
N	11·8	... ...	N	11·9
Cl	29·9	(calculated from nitrogen)	Cl	30·2
	99·8			100·0

The agreement with theory is sufficiently close to identify this compound as diamidotetrahydronaphthalene hydrochloride. From some uncrystallized nitro compound we obtained by the above methods an amido compound which went rapidly black, and was probably an isomer of the first, but was not analysed. The foregoing investigations having demonstrated the presence of decahydronaphthalene and tetrahydronaphthalene in the fractions experimented upon, we next attempted to prepare these substances synthetically.

Tetrahydronaphthalene was prepared by the action of sodium on a solution of naphthalene in amyl alcohol (according to the method of Bamberger and Kitschelt), the sodium being added gradually to the boiling solution. The oil obtained was separated, dried, and distilled. After many repetitions we finally obtained about 200 c.c. of distillate, boiling between 203° C. and 206° C. This was cooled to freeze out any naphthalene and redistilled. The distillate between 203° C. and 206° C. gave the following constants: Specific gravity, 0·977 at 15°, $N_D = 1{\cdot}5712$, $\frac{N_D - 1}{d} = 0{\cdot}584$.

Bamberger and Kitschelt give the gravity as 0·978, and the boiling-point at 205° C. The tetrahydronaphthalene obtained as above was next gasified at 1,260° F. with the following results:

C.c. of gas per c.c. of oil	134·5
Hydrocarbons	13·6
Valuation figure	1,829

The significance of these results will be referred to later. From the tetrahydronaphthalene we attempted to prepare decahydronaphthalene by passing its vapour together with hydrogen over reduced nickel, but without success, although by this method we had previously obtained a good yield of hexamethylene from benzene without difficulty. We finally prepared some decahydronaphthalene by treating tetrahydronaphthalene with hydriodic acid and phosphorus in sealed tubes. Eleven tubes, each containing 10 c.c. of tetrahydronaphthalene, 0·5 gram of phosphorus, and 25 c.c. of hydriodic acid, saturated at 0° C., were sealed off and heated in a bomb furnace. The first four were heated to 260° C. for thirty-six hours. As they gave a very poor yield, the remaining seven tubes were heated only to 210° C., and the time was extended to four days. From these tubes we obtained 14 c.c. of a liquid boiling between 170° C. and 178° C. This was redistilled, the greater portion coming over between 170° C. and 173° C. This fraction had the following constants

Specific gravity = 0·8426 at 15° C.
N_D ... ... = 1·4486 at 20° C.
$\frac{N-1}{d}$ ... ... = 0·532

		Theory—for $C_{10}H_{18}$
C ...	86·91	86·96
H ...	12·90	13·04
	99·81	100·00

The gravity and boiling-point are thus very slightly lower than those given by Wreden, and are in close agreement with the decahydronaphthalene obtained by us from Borneo oil.

According to Engler, when petroleum oils are dissolved in a mixture of alcohol and ether (equal parts) and cooled, the paraffins solidify out, and may thus be separated and estimated. Five representative oils were fractionated so as to obtain those portions which boiled between 300° C. and 350° C., with the exception of Borneo, of which the portion boiling between 280° C. and 340° C. was retained.

Each of these fractions was then mixed with about four times its bulk of alcohol and ether, and cooled to $-18°$ C. by means of liquid ammonia.

In the case of the American and Roumanian oils solids were obtained, which were separated off and pressed between blotting-paper. The Russian, Texas, and Borneo oils separated into two portions, and in each case the portion insoluble in the alcohol-ether mixture was separated off.

It was naturally thought that these portions would principally consist of paraffins and olefines, and they were cracked in the usual manner. The solid from the Roumanian oil had a gravity of 0·839 at 42° C., and an iodine absorption (Hübl) at 7·09 per cent. As this evidently contained some olefine it was treated with Nordhausen, and after treatment the gravity was 0·857 at 15° C., and the iodine absorption was nil. The refractive index at 27° C. was 1·4709, therefore $\frac{N-1}{d} = 0·549$. These figures corrected to gravity at 15° C. and N_D at 20° C. give $\frac{N-1}{d} = 0·553$.

The American solid portion had a gravity of 0·849 at 15° C. and N_D at 20° C. was therefore 1·4734 $\frac{N-1}{d} = 0·557$.

The iodine absorption was 17·2 per cent.

The following table gives the results obtained on gasification of the above:

	C.c. of Gas per c.c. of Oil.	Per Cent. Hydrocarbons.	Valuation Figure.
Roumanian ...	465·4	35·4	16,475
American ...	431·2	40·0	17,248
Texas	430·2	27·2	11,701
Borneo	342·7	26·4	9,047
Russian ...	425·3	38·9	16,544

The insoluble portion of the Borneo oil had a gravity of 0·9286 at 15° C.

According to Kraft the gravity of the higher paraffins at their melting-points approximates to 0·777.

We prepared undecane by Krafft's method from oil of rue. The oil was obtained from the London Essence Company, and our best thanks are due to Mr. Burgess, their chief chemist, for the trouble he took to procure us oil rich in ketones, and for information as to the best method to prepare them.

His method, with very slight variations, was used, and was carried out as follows: 240 c.c. of the oil of rue was mixed with 1 litre of a 40 per cent. solution of sodium metabisulphite. The mixture was kept at 70° C. on a water-bath for five hours, with vigorous mechanical stirring all the time. Eight lots of 4 grams each of sodium carbonate were added at intervals of fifteen minutes. At the end of the five hours the mass was allowed to stand overnight, and the liquid portion poured off. The crystalline residue was washed three times with ether, cooled to 0° C., and then drained and pressed between blotting-paper. To the residue an equal weight of sodium carbonate and enough water to cover the mass were added. The mixture was heated on a water-bath under a reflux condenser for some hours. The liberated ketone was separated off and the mass extracted with ether and the ethereal extract added to the ketone. The ether was distilled off and the ketone fractionated. The portion boiling between 223° C. and 225° C. had a specific gravity of 0·8266 at 20° C. and N_D 1·4271. Theory for $N_D = 1·4231$, and according to Beilstein the gravity is 0·8268 at 20·5° C. Several lots of methylnonylketone were prepared in the above manner, and the yield was in some cases as high as 50 per cent. of the oil of rue taken. Some of the ketone thus obtained was mixed with an equal bulk of chloroform and one-third more than the calculated weight of phosphorus pentachloride added gradually to the liquid through a reflux condenser.

When all the phosphorus pentachloride had been added, the contents of the flask were heated on a water-bath for a short time, and then cooled and poured into water. The chloride and chloroform were separated off and the chloroform dissipated on the water-bath; 12 c.c. of the undecylene chloride were then treated with 25 c.c. of hydriodic acid (2·0 specific gravity) and a little phosphorus in sealed tubes at 210° C. for forty-eight hours. The contents of the tubes were poured into sodium thiosulphate solution and the oil separated and distilled.

The greater part came over between 190° C. and 200° C. It was redistilled, and the portion boiling between 193° C. and 195° C. (except 754 mm.) gave the following constants:

Specific gravity - 0·7466 at 15°/15°—

N 1·41817 N_a 1·41620

$$\frac{N_D - 1}{d} = 0·560$$

According to Krafft the density is 0·7448 15°/4°, and the boiling-point 194·5° at 760 mm.

The calculated refractive index, according to Bruhl's formula, is N_a - 1·41254.

The above substance is therefore fairly pure undecane. The undecane was next cracked in the usual manner, with the following results:

	At 1260° F.	At 1400° F.
C.c. of gas at N.T.P. per c.c. of oil ...	492·4	544·4
Hydrocarbons	36·4%	33·8%
Valuation figure	17,923	18,400

As we were unable to separate olefines from the petroleum oil, we give the preparation and the results obtained from an olefine prepared from oil of rue.

Methylnonylketone prepared in the manner already described was converted into the carbinol by mixing it with amyl-alcohol and adding a considerable excess of sodium to the boiling mixture, using a reflux condenser. This operation occupied about three days. When the reaction was completed, water was added and the methylnonylcarbinol and amyl-alcohol separated. The amyl-alcohol was distilled off, and the carbinol remaining was then very gradually run on to an excess of phosphorus pentoxide in a flask provided with a reflux condenser and heated on a water-bath. The resultant mass was next extracted several times with ether, and after distilling off the ether the oil obtained was fractionated in a four-bulb Young's dephlegmator; the fraction boiling 191° C. to 193° C. gave the following constants:

Specific gravity 0·7735 at 15°/15°

N_D	1·43325	$\frac{N-1}{d}$	0·560
N_α	1·43148	,,	0·558
N_β	1·44198	,,	0·571

The $\frac{N_\alpha - 1}{d}$, calculated according to Bruhl's figures, is 0·558. According to different authorities, the boiling-point of undecylene varies from 192° C. to 196° C., and the gravities from 0·790 to 0·8398 at 0° C.

The density calculated from the formula proposed by Isidor Traube (*Ber.*, 1895, xxviii., 2924) is 0·765 ± 0·02, which is in close agreement with our results. A Hübl iodine absorption of the olefine gave 164·4 per cent. iodine absorbed, the theory for undecylene being 164·2 per cent. This oil was therefore fairly pure undecylene. The undecylene obtained was next gasified in the usual manner, with the following results, 15 c.c. being used for each experiment:

	At 1260° F.	At 1400° F.
C.c. of gas at N.T.P. per c.c. of oil ...	493·6	550·4
Hydrocarbons	30·8%	29·0%
Valuation figure	15,202	15,961

The analysis of the gas obtained at 1260° F. was as under:

	Per Cent.
Benzene vapours	2·4
Heavy hydrocarbons	28·4
Methane	45·2
Hydrogen	23·9
	99·9

In our first attempt to prepare undecylene we used amyl-alcohol and sodium amalgam for the preparation of the carbinol, passing in a stream of carbon dioxide all the time. The resultant oil was treated with phosphorus pentoxide in the manner before described, but the temperature was kept much higher (about 200° C.) by means of an oil-bath. Very little undecylene was obtained, but a series of fractions of the following composition :

B.P.		Carbon.	Hydrogen.
240° C. to 280° C.	... ...	86·0	13·6
280° C. to 350° C.	... ...	86·85	13·03
350° C. to 370° C.	... ...	86·55	13·17
370° C. to 375° C.	... ...	83·05	13·57

Although these fractions were further examined, no very definite conclusions could be drawn as to their constitution. The last compound is possibly a ketone of approximately the formula $C_{32}H_{64}O$, or $C_{32}H_{62}O$.

In order to institute a comparison between our method of valuation by means of cracking on the small scale and the results obtainable in practical working on the large scale, we append a series of tests carried out on the large scale with certain oils. These oils were used in a carburetted water-gas plant erected by the Economical Gas Apparatus Construction Company.

In every case all possible precautions were taken to obtain the best results, which were as follows :

Pennsylvanian Oil.

Laboratory valuation figure, 16,000.

Quantity of gas made, 391,000 cubic feet.

Amount of oil used, 969 gallons = 2·47 gallons per 1,000 cubic feet.

Average candle-power, 22·12 = 8·9 candles per gallon per 1,000 cubic feet.

Quantity of coke used in generator, 13,260 pounds = 33·9 pounds per 1,000 cubic feet.

Russian Oil.

Laboratory valuation figure, 15,927.

Quantity of gas made, 1,186,000 cubic feet.

Quantity of oil used, 3,078 gallons = 2·59 gallons per 1,000 cubic feet.

Average candle-power, 22·61 = 8·73 candles per gallon per 1,000 cubic feet.

Quantity of coke used in generator, 38,556 pounds = 32·5 pounds per 1,000 cubic feet.

Texas Oil.

Laboratory valuation figure, 11,697.

Quantity of gas made, 349,000 cubic feet.

Amount of oil used, 1,144 gallons = 3·277 gallons per 1,000 cubic feet.

Average candle-power, 20·51 = 6·2 candles per gallon per 1,000 cubic feet.

Quantity of coke used in generator, 12,240 pounds = 35·07 pounds per 1,000 cubic feet.

Borneo Oil.

Laboratory valuation figure, 8,024.

Quantity of gas made, 5,410,000 cubic feet.

Amount of oil used, 19,645 gallons = 3·63 gallons per 1,000 cubic feet.

Average candle-power, 17·03 = 4·69 candles per gallon per 1,000 cubic feet.

Amount of coke used in generator, 178,500 pounds = 33 pounds per 1,000 cubic feet.

Taking American oil as a standard, the following comparisons may be instituted:

	Laboratory Test.	Works Test.
American oil	100·0	100·0
Russian oil	99·5	98·1
Texas oil	73·1	70·3
Borneo oil	50·1	52·7

From the above, it is evident that our method of valuation yields approximately correct results.

Having now completed the experimental portion of our paper, we will proceed to discuss the conclusions which we think may fairly be drawn from the foregoing results.

A consideration of the figures in the distillation test tables makes it evident that, comparing fractions of the same boiling-point, the lower the gravity and refractive index, the better the oil for gas-making purposes.

The following table gives a summary of the constants obtained for the different classes of hydrocarbons:

Name.	Boiling-point, C.	Specific Gravity.	N_D	$\frac{N_D - 1}{d}$	Valuation Figure.
Undecane	194°	0·746	1·4182	0·560	18,400
Undecylene	193°	0·773	1·4332	0·560	15,961
Decahydronaphthalene ...	172°	0·843	1·4507	0·534	11,373
Tetrahydronaphthalene ...	205°	0·977	1·5712	0·584	1,829
Hexahydrocymene (according to Markownikoff) ...	161°	0·783	1·4323	0·552	

From this table it will be seen that, although the boiling-points are not far apart, the paraffin has the lowest gravity, the olefine, decahydronaphthalene, and tetrahydronaphthalene increase in gravity in the order named. The same also holds good for the refractive index, while, on the contrary, the value for gas-making purposes decreases in the same order. A further class of bodies, which we have not yet succeeded in preparing synthetically, consist of such compounds as hexahydrocymene. Such cycloparaffins partake of the nature of both open chain and ring compounds, and their value for gas-making purposes will no doubt depend on the relative number of carbon atoms contained in the chain and ring respectively.

The presence of such compounds is indicated by the high gravity of the solid paraffins separated from American and Roumanian oils, whilst the specific refraction is only slightly lower than that of normal paraffins. Generally we think we are justified in concluding from our results that—

1. The open chain compounds have the best value for gas-making purposes.
2. The presence of double bonds in the chain slightly decreases the value.
3. The presence of one or more rings diminishes the value considerably.
4. The more fully hydrogenated the ring, the better its value, as compared with other ring compounds.
5. Benzene rings have practically no value for cracking purposes.

Next, considering the relation of the gravity to the refractive index as expressed by the specific refraction, there are some important and interesting deductions to be made.

The open chain compounds, both paraffin and olefine, have a specific refraction of about 0·555 to 0·560. The presence of a hexamethylene ring lowers the specific refraction, and a saturated double ring reduces it still further. On the other hand, the specific refraction is raised considerably by the presence of an unsaturated ring, as in the case of tetrahydronaphthalene.

Having deduced these conclusions from an examination of the pure hydrocarbons, it will be well to consider their bearing on the constants given by some of the various gas oils examined. For this purpose it will be well to consider the oils from the different regions separately, and in most cases, for simplicity, to compare the fractions boiling between 280° C. and 300° C., as these are contained in all the oils examined.

In Pennsylvanian oil the gravity and refractive index of this fraction are lower than those of any of the other oils examined. The specific refraction (0·556) approaches that of a paraffin. This would indicate that the fraction contained a large proportion of paraffins and olefines. This opinion is modified by the fact that its gravity (0·833), and that of the solid portion obtained on freezing out from ether-alcohol solution (0·849), are too high in relation to the melting-point and boiling-point to consist entirely of these substances. They would, however, quite agree with a mixture of paraffins, olefines, and what we may term paraffinoid bodies—*i.e.*, compounds containing one or more long open chains, together with a hexamethylene ring.

Applying the same process of reasoning to the case of Russian oil, we should conclude that this oil consists principally of paraffinoid substances, as, though the gravity (0·862) is somewhat higher than in the case of American, the specific refraction (0·551) is rather lower. This conclusion is supported by the fact that it is difficult to freeze out any solid substance from the ether-alcohol solution, and that the figures obtained could not agree with any other conclusion or with the high valuation figure obtained.

In the case of Texas oil the gravity is 0·895, and is higher than that of either Pennsylvanian or Russian, and the specific refraction is still further lowered to 0·549. This oil, therefore, probably contains considerable quantities of complex ring compounds fully hydrogenated, but not tending to be paraffinoid in their nature. This is distinctly confirmed by the valuation figure.

The Roumanian oils vary considerably, but considering the average gravity (0·882) and the specific refraction (0·557) of the fraction previously indicated, we think that they must contain a considerable quantity of unsaturated ring compounds, as the gravity precludes any great amount of compounds with long open chains, and the high specific refraction is inconsistent with a preponderance of fully saturated ring compounds.

Some Roumanian oils, however, have a composition differing from the average given, and fall between Pennsylvanian and Russian oils in their properties.

The Borneo oil examined by us differs from any of the preceding oils, in that it contains a larger amount of unsaturated and partially hydrogenated ring compounds. This is shown by its very high gravity (0·936) in the specified fraction, together with ts high specific refraction (0·560). This conclusion is also entirely in accordance with the valuation figure.

Of the other oils examined, the Kansas, with a gravity of 0·850 and a specific refraction of 0·555, is somewhat inferior to Pennsylvanian for gas-making purposes, and in composition it is between Pennsylvanian and Texas, but more nearly approaches the former.

Californian probably contains a fair proportion of paraffins and paraffinoid bodies, but, in addition to these, its constants indicate a certain amount of incompletely saturated compounds, particularly in the higher fractions.

The only Galician oil we have examined was very similar in gravity to Russian oil, but had a higher specific refraction, thus indicating a somewhat higher proportion of open chain compounds, but less than in the case of Pennsylvanian.

The Grosny oils approach the Russian in character, but are somewhat inferior, probably due to the presence of more paraffinoid bodies and fewer true paraffins.

The Scotch gas oils are, of course, not true petroleum oils. From their low gravity, together with the specific refraction of 0·560, one would almost expect a better valuation figure than that obtained. By the method of formation, destructive distillation, one would naturally expect a considerable proportion of olefines, and also some substances containing benzene rings. These latter are probably paraffinoid in their nature, as otherwise the gravity would be much higher. We have not, however, had time to devote the same attention to Scotch oils as to the others, and hope at some future time to investigate them further.

We recognise that none of our deductions as to the composition of the various oils can be taken too dogmatically, as the number of possible hydrocarbons is so great, while the constants from which the deductions have been drawn are few in number. Considering the present state of our knowledge of the higher hydrocarbons, we think that the method of reasoning adopted will be found to give a useful index as to the relative value of oils used for gas-making purposes.

DISCUSSION.

Mr. L. MYDDELTON NASH asked whether, in addition to the valuation of the oils from the volumes of gas and the proportions of hydrocarbons yielded, any photometric tests of the oil-gas had been made. He had at one time had a good deal to do with the manufacture of oils of this kind, and had always regarded Borneo oil as the lowest in value for gas-making purposes, his experience thus agreeing with that of the authors in this respect.

Mr. Ross said that the carburetted water-gas in the works tests had been examined photometrically, but not the oil-gas made in the laboratory. Some other kinds of Borneo oil and the higher fractions of certain Borneo wax oils gave considerably higher valuation results. Some of them contained solid paraffins, and so were of low specific gravity.

Mr. W. J. Atkinson Butterfield thought that the laboratory figures were not quite on all-fours with the works results, because the former were arrived at without any knowledge of the nature of the hydrocarbons in the gas. Very often the gas containing 20 per cent. of hydrocarbons from one oil would have a very much higher illuminating value than the gas containing 20 per cent. of hydrocarbons, estimated in the same way, from another oil, owing to the different nature of the hydrocarbons in the two cases. The method proposed by the authors, therefore, could not be expected to afford an absolute criterion of the gas-making value of an oil. Nevertheless, it was a very useful auxiliary test, and he had himself used it for some time in a very similar form to that now described. He thought it would be an improvement to make the carbonizing apparatus rather larger, and to use a somewhat larger gas-holder, so that a portion of the gas might be available for photometric and even for calorific tests. These tests should preferably be made on a mixture of the oil-gas with coal-gas, because all kinds of oil-gas had not the same power of enriching coal-gas, owing to the differences which occurred in the hydrocarbons according to the temperature at which the gas was made and the kind of oil used. The authors had remarked that benzene rings were of no value, and perhaps in one sense that was the case; but, as a matter of fact, carburetted water-gas owed its illuminating power to hydrocarbons, which were, roughly speaking, those contained in 90 per cent. commercial benzol. It was true that benzol and hydrocarbons containing benzene rings were useless for cracking, but it was not altogether useless to make benzene and use the coal-gas as a carrier of it, for in that way a greater illuminating duty was obtained from the oil than by converting it wholly into a permanent gas, while the mixture of coal-gas and benzene vapour was sufficiently permanent for transmission to the consumer, so far as all ordinary degrees of enrichment of coal-gas were concerned.

Mr. Leather said that theoretically Mr. Butterfield was, no doubt, quite right in saying that the hydrocarbons absorbed by fuming sulphuric acid had somewhat different illuminating values, but from the practical standpoint of the working of the oils in carburetted water-gas it was found that the proportion of hydrocarbons absorbed corresponded very fairly with the photometrically determined illuminating power of the gas, while the working tests showed that—in the case, at any rate, of the four oils they had tried—the laboratory results corresponded closely enough with practical values for the ordinary valuation of an oil. When they said that the benzene ring was of no value, they did not refer to benzene itself, there being no actual benzene in the oils they used. What they had found was that, in the case of a hydrocarbon like tetrahydronaphthalene, containing one saturated or practically saturated benzene ring, a low valuation figure was obtained. Mr. W. Irwin, on cracking some portions of ordinary coal-tar oil (which was, practically speaking, creosote oil, consisting chiefly of hydrocarbons containing benzene rings), had found these to show such poor values as to be really useless for gas-making. As to the illuminating value of benzene they agreed, but if there were more than a certain quantity of benzene in the oil, the greater part of it would go into the tar instead of into the gas. As long as the gas had to come into contact with tar the quantity of benzene it would carry was strictly limited. With regard to Mr. Butterfield's suggestion that they should use a larger laboratory apparatus, and so obtain enough gas for a photometric test, they would need for this 10 cubic feet of gas as an absolute minimum, and it was questionable whether on such a scale the cracking temperature could be exactly maintained. In the apparatus they used at present the cracking temperature was absolute—*i.e.*, the whole of the oil was exposed and cracked at an exact temperature within 10 degrees. The retort was heated in the muffle up to the temperature at which cracking was commenced (say, about 1260° F.), the gas was turned off, and the oil run in. The experiment took from three to three and a half minutes, and during that time the temperature never varied more than 10 degrees, because the fireclay casing of the retort, being slightly hotter than the retort itself, gave up heat to the latter at about the same rate at which the heat was taken up in cracking. This was a rather important point, and anyone repeating their experiments would have to use a retort of the same size and apparatus of a similar description, and to see that the time taken for running the oil in was the same, or, at any rate, to see that the relationship between the various factors was such that the cracking temperature was constant. On a larger scale it would not be possible to insure this.

Mr. W. J. ATKINSON BUTTERFIELD agreed that it was advantageous to work at a uniform temperature, but thought that photometric tests might be made, as he had suggested, by mixing the oil-gas with coal-gas. Tests made in that way, too, would more nearly represent practical conditions. As to the comparison between the laboratory and works tests, the works values for two of the petroleum oils seemed to him extremely low. The value given for the Scotch oil also seemed low. He had generally found it to be quite as high as, or even slightly higher than, that of Russian or Pennsylvanian oil. He quite agreed with Mr. Leather's remarks with reference to tar having only a low value for gas-making.

Mr. J. H. B. JENKINS said that a good deal of oil-gas was made by railway companies and used for illuminating purposes after compression. The value of an oil was usually judged by multiplying the candle-power into the number of cubic feet of gas made from a given quantity of oil, and if such a figure could, either now or at some future time, be included in the authors' results, it would be very useful, inasmuch as it would enable a direct comparison to be made between their laboratory valuations and the methods of valuation ordinarily adopted in practice.

Mr. ROSS said that they could not do exactly as Mr. Jenkins suggested, but the factor he had mentioned was given in their works tests for the carburetted water-gas. He might mention that the exact composition of the oil-gas was not at present known, but that they had reason to believe that certain bodies occurred in it the presence of which, perhaps, had not been anticipated. Amongst these was, undoubtedly, a considerable quantity of *cyclo*-pentadiene. This they had succeeded in separating.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Methylene Blue as a Test for the Freshness of Milk. P. T. Müller. (*Archiv Hygiene*, lvi., 108; through *Pharm. Journ.*, 1906, vol. 77, p. 31.)—The conversion of methylene blue, by lactose and allied bodies, into a colourless compound may be utilized as a test for the freshness of milk. Two c.c. of milk are placed in a test-tube, 0·2 c.c. of a very dilute methylene blue solution is added, and a layer of liquid paraffin poured over the mixture. The test-tube is then placed in an incubator at a temperature of 37° C., and the discharge of the colour takes place after a varying period of time. Fresh milk usually shows the change after eight or ten hours, whilst milk that has been kept for some time reduces the methylene blue after a period varying from a few minutes to half an hour. W. P. S.

Determination of Fat in Cheese. M. Weibull. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xi., 736-738.)—The following method of applying the Göttlieb process to the determination of fat in cheese is described: From 2 to 3 grams of the cheese are placed in a graduated tube and heated with 10 c.c. of ammonia to a temperature of 75° C., with occasional shaking. In most cases the cheese dissolves readily, in which case the contents of the tube are cooled and 10 c.c. of alcohol are added. If the cheese does not go into solution, the heating is continued after the addition of the alcohol. To the cold solution are then added 25 c.c. of ether; the tube is closed with a cork and the mixture well shaken; 25 c.c. of petroleum spirit are now introduced, and, after again shaking, the tube and its contents are placed aside for one hour. An aliquot portion of the ethereal layer is then withdrawn, evaporated in a weighed flask, the residue of fat is dried for two hours at 100° C. and weighed. Instead of taking a portion of the ethereal solution, the whole of the fat may be extracted by shaking out the mixture several times with the ether-petroleum spirit mixture. W. P. S.

The Determination of Sodium Chloride in Egg-Yolk. L. and J. Gadas. *Ann. de Chim. anal.*, 1906, vol. 11, p. 249.)—The ordinary methods are stated to give too low results, and the following method is therefore recommended: 1 gram of the sample is covered with 12 grams of pure potassium nitrate, 2 drops of $\frac{N}{10}$ sodium hydroxide solution added, and the crucible gently heated, with precautions to prevent loss in the deflagration. The saline residue is dissolved in water, the solution exactly neutralized with nitric acid, and the chlorides determined by titration with $\frac{N}{10}$ silver nitrate solution. C. A. M.

Influence of the Oxidation of Ethyl Alcohol on the Maturing of Brandy and Wine. A. Trillat. (*Chem. Centr.*, 1906, i., 580; through *Pharm. Journ.*, 1906, vol. 77, p. 733.)—It is shown that ethyl, propyl, butyl, and amyl alcohols are readily oxidized to acetals, especially in the presence of ferric chloride or hydrochloric acid.

The author suggests that the maturing of spirits and wines is due partly to the formation of these acetals, which are highly aromatic substances, and partly to the formation of esters. A mixture of dimethylaniline and dilute sulphuric acid may be employed for detecting these acetals, a blue or green coloration being produced in their presence. W. P. S.

A Test for Bleached Flour. J. T. Willard. (*Bull. Kansas State Board of Health,* 1906, ii., 160.)—Flours which have been bleached by the action of oxides of nitrogen (*cf.* Analyst, 1906, 227) may be detected in the following manner: The reagent employed is prepared by heating 0·1 gram of α-naphthalamine with 20 c.c. of concentrated acetic acid, decanting the clear liquid and mixing it with 130 c.c. of dilute acetic acid. To this solution is added 0·5 gram of sulphanilic acid dissolved in 150 c.c. of dilute acetic acid. A small quantity of the flour is now shaken with a few c.c. of water, about 2 c.c. of the reagent are added, and the mixture allowed to stand for a short time. If nitrites be present, a distinctly pink coloration develops. The delicacy of the reaction may be increased by subjecting the flour-and-water mixture to centrifugal action, and then testing the clear aqueous liquid. It is possible that after a sufficient lapse of time the nitrites in the flour may become completely oxidized, but a sample exposed in a thin layer under a large bell-jar for nearly three months still reacted strongly. W. P. S.

The Detection of Rice-Flour in Wheat-Flour. G. Gastine. (*Comptes Rendus,* 1906, cxlii., 1207-1210.)—Two drops of a solution of a suitable dye-stuff are mixed on a cover-glass with a small quantity of the flour, the solvent evaporated at 28° C. to 30° C., and the residue dried at 50° C., then heated for some minutes at 110° C. to 130° C., and examined in Canada balsam under the microscope. After this treatment the hilum of the rice-starch granules is clearly shown, whilst in the case of wheat it is rarely very distinct. Isolated starch granules of rice are exceptional, but masses of the starch cells, with their characteristic symmetrical markings, are to be seen. The method is stated to be capable of detecting as little as 1 to 2 per cent. of rice-flour. Suitable dye-stuffs mentioned are aniline blue, certain cotton blues, blue C4B, Meldola blue, aniline green, methyl green, auramine, and aniline violets, the most suitable strength of the solution being 0·05 gram in 100 c.c. of 33 per cent. alcohol. The dye-stuffs are not essential, they only render the markings more distinct. Good results can also be obtained by heating the flour without dye-stuff, and then treating it with osmic acid, silver nitrate (with exposure to sunlight), or gold chloride. The method is also satisfactory with maize and buckwheat flour, the starch granules of which behave like rice. The hilum in the cells of potato or arrowroot starch is also made clearly visible in the same way. Unlike most starches, these last are dyed by contact with the solutions of the dye-stuffs. C. A. M.

The Determination of Malic and certain other Fixed Acids in Fruit-Juice. W. Mestrezat. (*Comptes Rendus,* 1906, cxliii., 185, 186.)—This method, which is stated to be specially applicable to musts and wines, is based upon the fact that the malate, tartrate, and succinate of barium are insoluble in alcohol of 75 per cent. strength, whilst the barium salts of the other acids (lactic, glycollic, etc.) that may be present remain in solution. A known volume of the juice is neutralized with barium hydroxide, and then rendered slightly acid by the addition of 3 or 4 drops of 3 per cent. acetic acid, and concentrated to 15 c.c. by boiling for a few minutes *in vacuo*. It is next treated with 2 c.c. of a 30 per cent. solution of barium acetate, and sufficient alcohol added to bring the alcoholic strength to about 80 per cent. The insoluble substances are collected, washed with 80 per cent. alcohol, and then taken up with water (about 12 to 15 c.c.) containing sufficient sulphuric acid to liberate the acids from the barium salts. The magma is diluted to a definite volume, say 100 c.c., with concentrated alcohol, the gums, pectin derivatives, and proteids being precipitated, whilst the acids and tannins remain in solution. The tartaric acid is precipitated by adding potassium chloride and potassium acetate to 80 c.c. of the filtrate, then diluting the liquid to 100 c.c., and allowing it to stand for forty-eight hours at 15° C. The precipitated hydrogen potassium tartrate is filtered off and washed with alcohol of 65 per cent. strength, whilst the filtrate will only contain traces (0·01 gram per 100 c.c.) of tartaric acid, for which an allowance can subsequently be made. The filtrate is next neutralized with a saturated solution of barium hydroxide, rendered faintly acid as before with acetic acid, and brought up to 80 per cent. alcoholic strength by the addition of strong alcohol. The precipitate is dissolved in water slightly acidified with hydrochloric acid, and yields a solution containing only malic and succinic acids, plus tannins, which can be completely separated by Laborde's aceto-mercuric reagent. The malic acid is determined by titration with potassium permanganate, in the presence of sulphuric acid, after removal of all traces of alcohol. After oxidation of the malic acid the liquid is evaporated to dryness with sand, and the succinic acid extracted with ether from the dry residue. This method is stated to give absolutely concordant results, and in test experiments in which the three acids were added to organic liquids, the theoretical quantities were recovered. Sugar and glycerin have no influence on the determination. C. A. M.

The Determination of Tartaric Acid in Wine Lees and Tartrates. P. Carles. (*Bull. Soc. Chim.*, 1906, xxxv., 571-575.)—Various precautions required to obtain accurate results in the method of Goldemberg and Géromon are embodied in the following modification, devised by the author. It is also shown that the presence of calcium phosphate has no appreciable influence, but that iron phosphate causes a loss of about 1 per cent. of tartrate, and that the presence of aluminium may increase this loss to 2 per cent. In the new method 6 grams of wine lees or 3 grams of crude tartrate are finely powdered and digested for an hour with 9 c.c. of dilute hydrochloric acid (specific gravity 1·10), the flask being shaken from time to time. An equal amount of water is then added and the digestion continued for another hour, after which the contents of the flask are made up to 100 c.c., thoroughly shaken, and filtered. Fifty c.c. of the filtrate are poured into a beaker already containing 18 c.c. of a 20 per cent. solution of potassium carbonate, and the mixture gradually heated, and finally boiled for fifteen to twenty minutes, the vessel being meanwhile kept covered. The liquid is now filtered off with the aid of suction, the filter washed with boiling water, and the filtrate and washings evaporated on the water-bath until not less than 13 c.c. or more than 15 c.c. are left. It is next left to cool for fifteen minutes, after which 3 to 4 c.c. of glacial acetic acid (specific gravity 1·064) are added and the mixture shaken for ten minutes, and then mixed with 100 c.c. of alcohol of not less than 94 per cent. strength. The precipitate becomes granular and crystalline after being stirred for five minutes, and the supernatant alcohol can be decanted on to a filter. The residue is washed by decantation about a dozen times with fresh portions of 10 c.c. of alcohol, the washing being then completed on the filter with the aid of suction until the washings are neutral. Finally the filter and precipitate are boiled with 100 c.c. of water for a minute, and the solution immediately titrated with standard sodium hydroxide solution with phenolphthalein as indicator. For wine lees the ordinary correction is made—viz., for a yield of 20 per cent. a deduction of 0·70, and for a yield of $20+n$ per cent. a deduction of $0{\cdot}70+(n\times 0{\cdot}2)$. This correction is not required in the case of tartar and crude tartrates. C. A. M.

Starch in Powdered Capsicum. J. Hockauf. (*Zeits. Oesterr. Apoth. Ver.*, 44, 303; through *Pharm. Journ.*, 1906, vol. 77, p. 106.)—Powdered capsicum is frequently mixed with about 1 per cent. of fixed oil to improve its appearance, and such oiled powders often contain more or less maize starch, etc. The starch in these samples may be detected, without previous removal of the oil, by mixing a little of the powder on a slide with an alcoholic solution of iodine, and then adding a solution of chloral hydrate. When examined under the microscope, many of the oil globules will appear dark blue, and remain so for half an hour. After the preparation has become quite clear, a little more chloral hydrate is added. In this way the various tissues are shown clearly, and the starch now appears pale blue. W. P. S.

The Differentiation of Pharmaceutical Benzoic Acids. Cormimbœuf and L. Grosman. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 243, 244.)—Benzoic acid prepared from gum benzoin has a greater commercial value than benzoic acid prepared synthetically from benzyl chloride, and the former is therefore frequently substituted for the latter. A simple method of differentiating the two products is to dissolve a few grams in hot water containing sodium carbonate, when the benzoin acid emits a characteristic pleasant odour, whilst the synthetic acid gives off a quite different odour, recalling that of parsley. The latter odour can be masked, however, by perfuming the benzoic acid with, *e.g*, vanillin, or by subliming it with a little gum benzoin. Another test is based on the fact that the synthetic acid always retains a certain proportion of organic chlorine, which can be detected by igniting 5 grams of the sample with an equal quantity of sodium carbonate, extracting the residue with hot water, acidifying the filtrate with nitric acid, and adding silver nitrate. Benzoic acid from benzoin gives only an insignificant reaction, whilst the synthetical product shows unmistakably the presence of chlorine. C. A. M.

An Adulteration of Oil of Cade. C. Pépin. (*Journ. Pharm. Chim.*, 1906, xxiv., 49-58.)—Oil of cade is stated to be frequently adulterated with a variable proportion of pine-tar oil. The addition may be detected by shaking 1 c.c. of the sample with 15 c.c. of petroleum spirit, filtering the extract, and shaking 10 c.c. of the filtrate with 10 c.c. of a 5 per cent. solution of neutral copper acetate. The mixture is allowed to stand, and 5 c.c. of the petroleum spirit layer decanted, mixed with 10 c.c. of ether, and filtered. Should the cade oil have contained pine-tar oil, the liquid will have an intense green colour, whereas a pure product will give only a light brownish-yellow tint. This reaction is probably due to the formation of green copper resinates soluble in petroleum spirit from the resin acids in the pine-tar oil. Hirschsohn asserted that a similar reaction was given by pure oil of cade, but the authors have never found this to be the case with samples of known purity obtained by distillation from *Juniperus oxycedrus* alone. In their opinion every oil giving the reaction ought to be rejected, though the absence of a green coloration does not necessarily mean that the oil is genuine. The test is capable of detecting 10 per cent. of pine-tar oil, and a smaller addition would not pay. C. A. M.

Characteristics of Essential Oil of Rue. H. Carette. (*Journ. Pharm. Chim.*, 1906, xxiv., 58-62.)—It is commonly accepted that commercial oil of rue is the product of *Ruta graveolens*, and that it contains about 90 per cent. of methylnonylketone. It was shown, however, by Soden and Henlé, and subsequently by Lees and Power, that oil of rue from Algiers contained a large proportion of methylheptylketone. The author was unable to detect this compound in French oil from *R. graveolens*, and has therefore examined the Algerian products. An oil known in Algiers as *essence de rue d'été*, is obtained from *R. montana*. This consists, in the main, of methylnonylketone, and therefore solidifies readily in the winter. Its solidification point (+5° to +8° C.) approximates closely to that of the oil from *R. graveolens* (+8° to +10° C.). Another product, termed *essence de rue d'hiver*, is distilled from *R. bracteosa*, and its properties differ greatly from those of the oils from *R. montana* and *R. graveolens*. It does not solidify in winter, its solidification-point being about −18° C., and it does not become liquid again until warmed to about −10° C. It combines almost entirely with sodium bisulphite, the combining substances consisting chiefly of methylheptylketone, whilst methylnonylketone is only present in small quantity. All these different essential oils of rue are soluble in 2 to 3 parts of 70 per cent. alcohol, whilst the presence of oil of turpentine or petroleum spirit prevents the formation of a clear solution. The isolation of the ketones and determination of their melting-points will give valuable indications as to the origin and quality of a given oil of rue. C. A. M.

Separation of Brucine and Strychnine by Treatment with Nitric Acid. W. C. Reynolds and R. Sutcliffe. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 512-515. —An examination of the various modifications of this process proves that the conditions proposed by both Stoeder and Gordin give slightly more accurate results than Keller's original process. The conclusions arrived at are—(1) that for an amount of total alkaloid up to 0·4 gram the reacting solution should contain at least 7 per cent. of HNO_3; (2) that the oxidation of the brucine is complete after ten minutes, when the reaction should be stopped; (3) that the temperature should not exceed 25° C.; (4) that excess of caustic soda or potash should be used to liberate the strychnine, not sodium carbonate or ammonia; and (5) that nitric acid of specific gravity not less than 1·42 should be used; otherwise the addition of a trace of nitrite may be necessary to start the reaction (*c.f.* ANALYST, xxx., 261). W. H. S.

Determination of Cantharidin in Cantharides. K. Siegfried. (*Schweiz. Wochenschr.*, xliv., 342; through *Pharm. Journ.*, 1906, vol. 77, p. 9.)—The following method is proposed and is a modification of that devised by Panchaud for the Swiss Pharmacopœia: Fifteen grams of the finely-powdered cantharides are placed in a 250 c.c. Erlenmeyer flask, to which are added 150 grams of chloroform and 1 c.c. of hydrochloric acid of a specific gravity of 1·124. The mixture is shaken for several minutes, and then allowed to stand for twenty-four hours, with occasional shaking. After filtration through a covered filter, 100 grams of the filtrate are evaporated at as low a temperature as possible on the water-bath, and the residue treated with 10 c.c. of petroleum spirit. The crystals are collected on a weighed filter, washed with a little petroleum spirit, and dried at a temperature of 50° C. W. P. S.

Examination of a Sample of Eucalyptus Oil, an Overdose of which caused Death. F. A. U. Smith. (*Pharm. Journ.*, 1906, vol. 77, p. 662.)—Whilst the relative therapeutic values of the two classes of eucalyptus oil, known respectively as the cineol oils and the phellandrene oils, is not at present precisely known, the Pharmacopœia gives preference to the former class of oil, the oil of *Eucalyptus globulus;* but the latter class, as typified by the oil of *E. amygdalina*, was official until the year 1898. A case of poisoning by an overdose of the oil having occurred at Derby, the author examined the oil with a view of ascertaining whether it was composed chiefly of cineol or phellandrene. The results obtained were: Specific gravity, 0·919 at 15° C.; rotation in 100 mm. tube, + 4·47°; cineol, 52·65 per cent.; phellandrene, absent. The deceased took about half a wineglassful with a similar quantity of warm water. W. P. S.

ORGANIC ANALYSIS.

Influence of Dilution and the Presence of other Reducing Sugars on the Osazone Test for Glucose and Fructose. H. C. Sherman and R. H. Williams. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 629-632.)—In pure glucose solutions, tested at constant volume with fixed amounts of phenylhydrazine hydrochloride and sodium acetate, the time required for the precipitation of the osazone varies with the amount of glucose present, and is nearly constant for any given dilution. In solutions containing only about 0·1 per cent. the time required for precipitation is much reduced by the presence of 1 per cent. or more of sucrose, but only slightly by the presence of 5 per cent. of raffinose. Solutions of fructose and invert sugar show similar variations with concentration, those of fructose always yielding a precipitate of osazone in about one-third the time required for the same amount of glucose, and those of invert sugar in slightly more than one-third the time. Sucrose also slightly accelerates the osazone precipitation in dilute solutions of fructose.

Maltose retards the precipitation of glucosazone, interfering much more seriously in the case of glucose than with fructose, and lactose interferes similarly, but to a greater degree. W. H. S.

Esterification as a Cause of Error in the Determination of Sugar. Talon. (*Ann. de Chim. anal.*, 1906, vol. 11, pp. 244, 245.)—If alcohol is present in a mixture in which cane-sugar is to be determined by inversion, it is necessary to remove the alcohol to prevent the formation of esters. In the case of dextrose, ethyl-dextrose, a non-reducing body, is formed. The esterification increases with the time and temperature, and in the presence of concentrated alcohol the yield may amount to nearly 100 per cent. The esterification of dextrose and levulose ceases when the proportion of alcohol is less than 40 per cent., but an error is still produced in polarimetric determinations. In the absence of an acid no esterification takes place. Methyl-alcohol acts in a similar manner, and glycerin also forms esters with dextrose and levulose; but the small amount of glycerin in fermented alcoholic liquids has no influence on the determination of sugar. C. A. M.

Optical Rotation and Density of Alcoholic Solutions of Gliadin. W. E. Mathewson. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 624-628.)—The specific rotation of gliadin in 70 to 75 per cent. alcohol is practically independent of the gliadin concentration, but in 70 to 80 per cent. alcohol decreases with increase in the alcoholic concentration. It increases slightly with increase of temperature between the limits 20° C. to 45° C. The variation in density of gliadin solutions for such concentrations as would be met with in flour analysis is so small as to render even an approximate determination of gliadin in alcoholic solution by means of the density extremely difficult. W. H. S.

On the Determination of Petroleum, Petroleum Distillates, and Benzene in Oil of Turpentine and its Substitutes. Richard Böhme. (*Chem. Ztg.*, 1906, xxx., 631.)—In a long paper the author compares and criticises a number of methods for the detection of adulterants in oil of turpentine, and also communicates a table containing the results obtained by him in the examination of a number of samples. He has modified Herzfeld's sulphuric acid method in order to save time, and works as follows: Twenty c.c. of a mixture of 1 volume of fuming and 3 volumes of concentrated (1·84) sulphuric acid are placed in a specially constructed flask which holds about 40 c.c. up to its neck, the latter holding an additional 10 c.c. and being graduated in $\frac{1}{5}$ c.c. To the acid 10 c.c. of the oil to be examined are slowly added; after cooling, the whole is well mixed and allowed to stand in the stoppered flask for one hour; concentrated sulphuric acid is then cautiously added until all the unattacked oil has been brought into the neck of the flask, which is then allowed to stand for another two to three hours, after which time the volume of the residual oil is read off. Occasionally the flask must be allowed to stand for six hours before the volume is read. The author finds that pure oil of turpentine treated in this way will give a residue of about 0·9 c.c., but sometimes even 1·6 c.c. are found; the refractive index of this, however, is higher than that of the original oil. In mixtures of oil of turpentine with petroleum products, a residue of 1·25 c.c. corresponds to an addition of about 5 per cent. of petroleum, 1·70 to 10, 2·00 to 15, 2·50 to 20, 2·80 to 25, 3·35 to 30, 3·75 to 35, 4·15 to 40, and 4·50 c.c. to 45 per cent. If a residue of 4·5 to 5·5 c.c. is obtained, the test should be repeated, using 20 c.c. of ordinary concentrated acid; if more than 5·5 c.c. is found, the test is again carried out with only 10 c.c. of ordinary acid. The percentage of petroleum is obtained by multiplying the volume of residue found by 10. If benzene is the adulterant present, it must be remembered that a considerable proportion of a commercial benzene is attacked by sulphuric acid, especially if the temperature be allowed to rise; hence the results obtained are not so reliable.

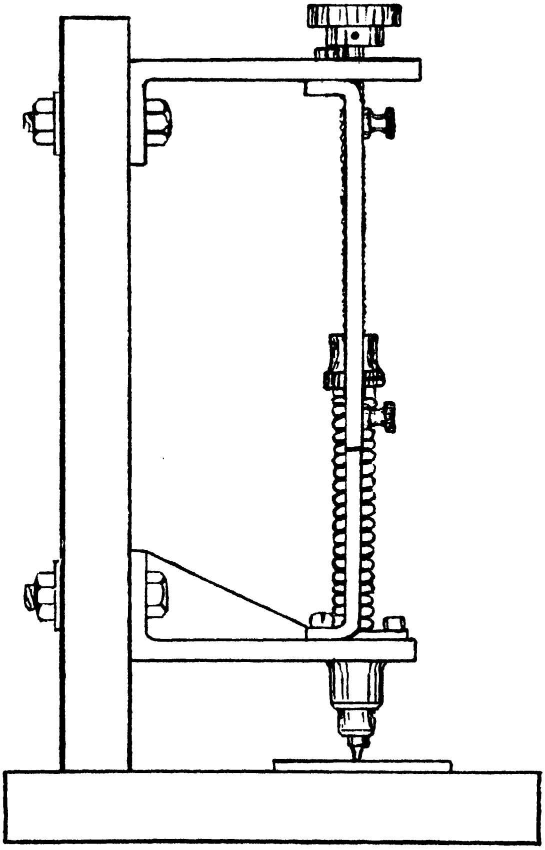
Commenting on the above paper, Herzfeld (*Chem. Ztg.*, 1906, **xxx.**, 697) mentions that he now carries out his method in a separating funnel graduated in $\frac{1}{10}$ c.c. instead of in a burette. For the detection of small quantities of benzene, the oil should be fractionated, and the different fractions obtained examined. In doubtful cases the residual oil left after treatment with sulphuric acid is tested by Burton's nitric acid method, as well as by the refractometer. A. G. L.

The Reduction of Cinnamic Acid to Cinnamene by Mould Fungi. Oliviero. (*Journ. Pharm. Chim.*, 1906, xxiv., 62-64.)—The enzymes secreted by *Aspergillus niger*, *Penicillium glaucum*, and possibly by other mould fungi, are capable of effecting a rapid reduction of cinnamic acid to cinnamene. Thus a cultivation of *A. niger*, grown on Raulin's fluid until complete development of the mycelium, and then thoroughly shaken and filtered through a Chamberland filter, yielded a clear liquid, which, when shaken with a weak solution of sodium cinnamate, immediately gave the characteristic odour of cinnamene. The reaction is sufficiently sensitive for cinnamic acid to be used as a reagent for the detection of these mould fungi in medicinal preparations and food products. It affords the explanation of the occasionally rapid deterioration of pharmaceutical products, such as balsam of Tolu, which are rich in compounds of cinnamic acid. C. A. M.

Muskone, the Aromatic Constituent of Musk. H. Walbaum. (*Journ. Prak. Chem.*; through *Chem. Zeit.*, xxx., 194, and *Pharm. Journ.*, 1906, vol. 77, p. 31.)—The odoriferous constituent of musk, muskone, is present in musk in only very small proportion. It is a viscid, colourless oil, very slightly soluble in water, but readily soluble in alcohol. It has a powerful and agreeable odour of musk, and, like ionone, rapidly exhausts the nerves. By steam distilling 500 grams of musk for several days, the author obtained 1·4 per cent. of a brown oil, and on redistilling this under a pressure of 9 mm., about one-half came over between 200° C. and 210° C. as a yellowish-brown viscid oil. After warming the latter with an alcoholic solution of potassium hydroxide, it distilled under 7 mm. pressure at 160° C. to 164° C.; the distillate gave with semicarbazide a crystalline carbazone, from which the oil was regenerated by sulphuric acid. Muskone is, therefore, a ketone. W. P. S.

Determination of Total Soluble Bitumen in Paving Materials. S. Avery and R. Corr. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 648-654.)—The method proposed by the authors consists in extracting with carbon bisulphide in a Soxhlet extractor a weighed quantity of the material contained in a capsule of hardened filter-paper. The extraction is continued until the solvent has siphoned over colourless some ten times, the solvent with the extracted bitumen ignited in a platinum dish, and the percentage of residue determined. The filter-paper and its contents are dried at 110° C., cooled in a weighing-bottle, and weighed, the loss in weight less the material recovered from the solvent representing the amount of dissolved bitumen. The results thus obtained for Bermudez and Malthas asphalts agree closely with those given by the methods of Dow and Richardson, in which the solution is filtered through asbestos or by Richardson's centrifugal method. The practice sometimes adopted of igniting the filter-paper apart from its contents, after extracting the bitumen, and adding the ash to the residue before weighing, is shown to give distinctly too high results. W. H. S.

A Method of Testing the Hardness and Elasticity of Varnishes. A. P. Laurie and F. G. Baily. (*Journ. Roy. Scot. Soc. Arts*, 1906, xvii., 101-111.)—The authors have devised a simple apparatus to determine the degree of pressure required to make a scratch upon a film of the given varnish on a glass plate, thus obtaining a numerical value for the hardness and toughness. The apparatus consists of a central rod moving easily in a vertical direction through openings in two supporting brackets. On the upper part of the rod is a screw thread on which is a running nut, prevented from turning by guides which project into the wings of the nut, whilst by turning a milled head at the top the rod is made to revolve and the nut to travel up or down. A helical spring is fixed at one end to the lower part of the nut and at the other to the lower bracket, whilst the lower end of the rod carries a blunt steel point. Thus by turning the milled head at the top the point can be raised or pressed down on to the plate. A scale is attached to one of the guides of the running nut, and the position at which the point is just at the height of the film is taken as zero. Fig. 1 on the scale represents 0·1 kilo, No. 2, 0·2 kilo, and so on. The point is rounded to a spherical surface and has a radius of 1 mm. The method of using the instrument is to draw



the varnished plate slowly under the point, the pressure being meanwhile steadily increased until a white scratch becomes visible. No notice is taken of grooving, which may even be regarded as a sign of toughness in a varnish. The spring used by the authors gave a maximum pressure of 1,200 grams, but few of the best varnishes required this pressure. Duplicate determinations by different observers differed from one another by not more than 5 per cent. The films of varnish were prepared by cleaning the glass plate, polishing it with leather, and placing it in the water-oven with the sample of varnish. The varnish was then poured on the centre of the plate and distributed with the finger, after which the plate was warmed on the top of the oven so that the film became level and uniform, and was finally placed in a cupboard with levelled shelves. Experiments were made with varnishes prepared from common rosin and oil and from gum animi, and kauri and white Manilla copals, and with representative commercial varnishes. The following table gives typical results obtained in determinations of the rate of drying:

Varnish.	One and a Half Months.	Two and a Half Months.	Four Months.	Eight Months.	Nine Months.	Twelve Months.
Rosin and oil	—	—	1·2	1·7	1·9	1·9
Picture copal	—	—	6·5	8·0	9·0	9·5
" "	—	—	3·5	4·0		6·0
Hard church oak ...	—	—	4·8	6·5		12·5
" "	—	—	1·5	4·5		9·0
Carriage	—	—	5·5	7·0	—	12·0
Finest oak	—	—	5·5	8·0	—	10·0
Maple	—	—	6·5	8·5	—	—
Amber	—	—	1·0	1·0	—	1·0
Floor	4	4·5				
"	6	6·5				
Carriage	4	10·0				

Experiments were also made to determine the effect of exposure to air and frost upon different vanishes. It was found that an inferior copal varnish and the amber varnish showed signs of deterioration after a fortnight, and had disappeared in three months. The hard church oak varnishes had cracked in three months, whilst the other trade varnishes, though apparently sound and transparent, had decreased in hardness to one or two, and gave a crumbling scratch. Hence the test is capable of detecting decay of a varnish before the appearance of outward signs. C. A. M.

Electrical Method for the Simultaneous Determination of Hydrogen, Carbon, and Sulphur in Organic Compounds. H. N. Morse and C. W. Gray.

(*Amer. Chem. Journ.*, 1906, vol. 35, pp. 451-458.)—An extension of Morse and Taylor's method (ANALYST, xxx., 321) to include the determination of sulphur, the apparatus employed being, with slight modification, similar to that previously described. The coils in the vicinity of G are wound tightly about the porcelain tube to make room for a long platinum boat (F), in which is placed a weighed quantity of pure PbO_2 for the absorption of the SO_2 produced, the rear of the boat being open to facilitate contact between the products of combustion and the oxide. Asbestos plugs (J J) replace the rolls of oxidized copper wire gauze which formerly preceded and followed the boat K. For the complete absorption of nitrogen oxides, a glass tube (A), filled with asbestos covered with PbO_2, is inserted between the combustion-tube and the usual absorption apparatus for water and carbon dioxide, and maintained at 175° C. by surrounding it with an electrically-heated graphited porcelain tube (B) about 300 mm. long, and provided with an asbestos covering (C). Fig. 2 shows the complete apparatus set up for use. In making an analysis the boat containing the weighed quantity of peroxide is inserted at F, and that containing the substance to be analysed at K, the two circuits, one through the combustion apparatus, the other through ABC, closed, and dry air passed through the apparatus. After drying out completely, the absorption train is joined to A, the current of dry air continued, oxygen admitted at D, and the substance in K and the plug of asbestos in front of the boat heated by a lamp. The contents of the boat F after a combustion consist of a mixture of PbO_2, $PbSO_4$, and, if the substance contained nitrogen, a small quantity of $Pb(NO_3)_2$. The quantity of PbO yielded by unit weight of the peroxide is estimated before commencing the combustion, so that the amount of PbO in the boat after the combustion is known, and the excess over this, after reducing the peroxide and nitrate to oxide by heating in a current of air or nitrogen, gives the amount of sulphur as SO_3. The method has given satisfactory results with sulphonal and other organic compounds.

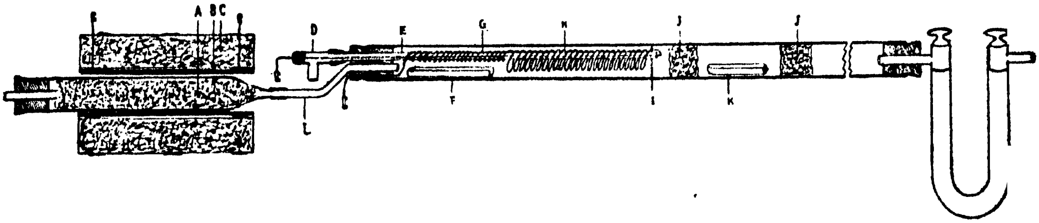


FIG. 1.

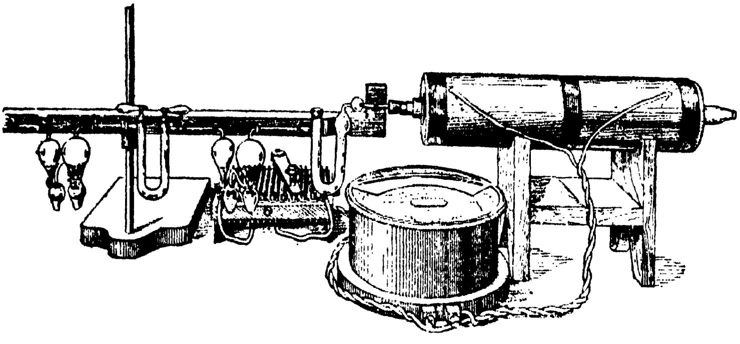
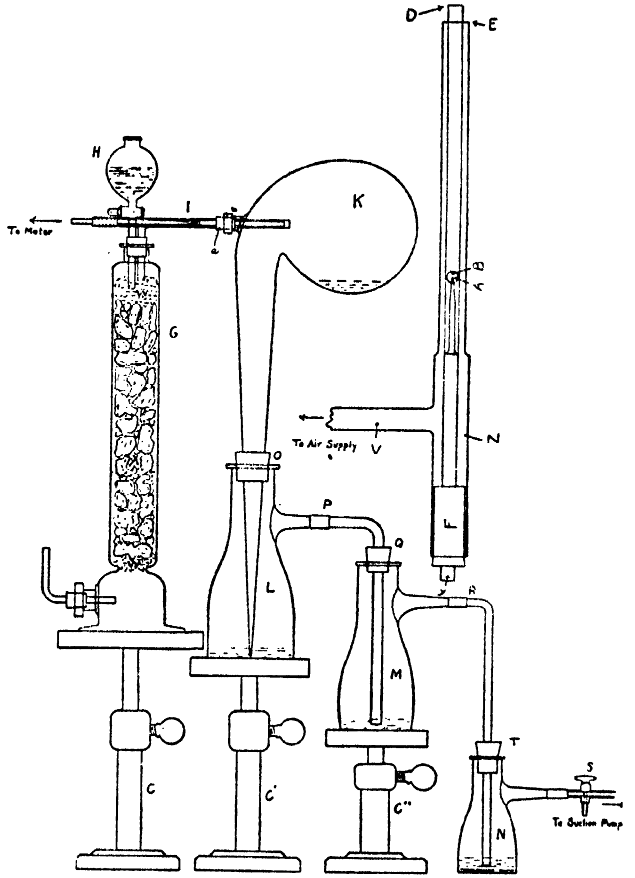


FIG. 2.

W. H. S.

Determination of Total Sulphur in Coal Gas. E. P. Harding. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 537-541.)—A modification of Drehschmidt's method is proposed, in which the gas is burnt in the presence of bromine vapour, the sulphur dioxide being thereby immediately oxidized, in the presence of water of combustion, to sulphuric acid, which is then aspirated through a solution of K_2CO_3, and the sulphur precipitated and weighed as $BaSO_4$. The apparatus employed is shown in the figure, in which G is a purifying tower, 45 cm. high and 6·3 cm. in diameter, filled with pumice-stone saturated with a $33\frac{1}{3}$ per cent. solution of KOH and contained between glass-wool layers (WW); H is a dropping funnel for delivering continuously the potash solution; and K a retort, 24 cm. deep, 16 cm. in cross-section, with a delivery tube 46 cm. long, fitted into the flask L by the rubber stopper O. L, M, and N are heavy glass suction bottles, with the respective diameters 10, 8·9, and 6·3 cm., connected at P and R with rubber tubing, and attached through the glass tap S with the suction pump. C, C′, and C″ are adjustable wooden supports. The burner I is connected at *y* with the meter, and at V, by means of a short piece of rubber tubing, with the purifying tower. It is of hard glass, and is fitted into the tubule of the retort with the rubber stopper *a*. The air-supply tube E is 9 mm. in diameter at E and 12 mm. at Z, and D, the gas-supply tube, is 5·8 mm. in diameter. The latter has opposite holes B for admission of air, and is drawn out at A to an opening sufficiently small to deliver, under aspiration, 0·35 to 0·5 cubic foot of gas per hour.



The burner is first connected with the meter and tower, the gas turned on, ignited, and allowed to burn for fifteen minutes, the retort and absorption flasks being meanwhile cleaned and partly filled with a 5 per cent. solution of K_2CO_3, 30 c.c. each being put into N and M, 50 c.c. into L, and 30 c.c. together with 4 c.c. of bromine into the retort K. The flasks and retort are then connected up, the gas turned off, the burner inserted into the tubule of the retort, and air aspirated through the apparatus for ten minutes. Aspiration is then momentarily discontinued, the burner removed from the retort, the gas turned on and ignited, the burner reinserted and the aspiration continued and regulated with the cock S. After 1½ to 2 cubic feet of gas have been burned, at the rate of 0·35 to 0·5 cubic feet per hour, the gas is turned off, the aspiration being continued until the apparatus has cooled down to room temperature. The burner is then withdrawn from the retort and rinsed with water into a 500 c.c. beaker, the retort and absorption flasks disconnected, their contents poured into the beaker, and these rinsed several times with small quantities of water. The contents of the beaker are acidified with HCl, concentrated to 100 c.c., transferred to a No. 3 beaker, and the sulphur precipitated and weighed as $BaSO_4$.

C. D. Jenkins (*ibid.*, 1906, xxviii., 542-544) describes a new and easily portable form of apparatus for the purpose, consisting of a system of condensers, at the bottom of which 0·8 to 1 cubic foot of gas, measured through a small dry meter, is burned in an ammoniacal atmosphere at the rate of 0·6 cubic foot per hour. The sulphur compounds form sulphur dioxide and sulphur trioxide, which, combined with the ammonia, are dissolved in the condensed water and collected, the apparatus being washed out with 200 c.c. of distilled water. To this solution are added 2 to 3 c.c. of bromine water, the liquid concentrated to 30 to 40 c.c., and an excess of a hydrochloric acid solution of barium chromate added. The whole is then gently boiled, an excess of dilute ammonia added, again boiled for a minute, filtered, and washed. The ammonium chromate in the filtrate, after being boiled in a stout flask with a Bunsen valve to expel air, is cooled and titrated direct with stannous chloride, using the iodo-starch blue to accentuate the end-point. The chromic acid found is equivalent to the sulphuric acid in the original solution. W. H. S.

INORGANIC ANALYSIS.

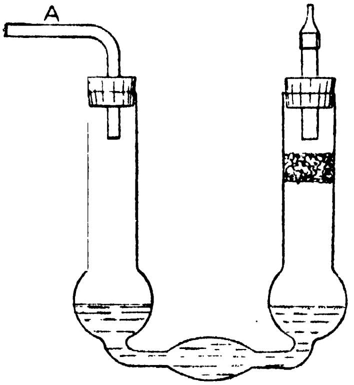
Rapid Method of Babbitt Metal Analysis. H. Yockey. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 646-648.)—One gram of filings or borings is treated in a small beaker with 20 c.c. of nitric acid (specific gravity 1·42) and water (1 : 2), placed on the water-bath and covered with a watch-glass until all action has ceased. It is then evaporated to dryness and baked in an air-bath at 120° C. for one hour. The residue is moistened with concentrated HNO_3, 30 to 40 c.c. of hot water added, the whole boiled vigorously for five minutes, and the mixed oxides of tin and antimony filtered off, washed, dried, ignited in a porcelain crucible, and weighed as $SnO_2 + Sb_2O_4$, any metal reduced in burning the filter being oxidized with a few drops of concentrated HNO_3.

The filtrate is diluted to 250 c.c. in a calibrated flask, and 50 c.c. pipetted out for estimation of lead, to which is added 10 c.c. of ammonia (1 part ammonia, specific gravity 0·9, to 1 part water) and 6 c.c. glacial acetic acid, the mixture heated to incipient boiling, and the lead titrated with a solution of ammonium molybdate, 1 c.c. of which = 0·01 gram lead, and standardized against pure lead dissolved in HNO_3, a 1 in 300 solution of tannic acid being used as indicator. To the remaining 200 c.c. of the solution is added sodium carbonate till a precipitate forms, then 1 or

2 c.c. of ammonia. After well stirring and allowing to settle, any copper present is titrated with standard KCN solution (1 c.c. of which = 0·0025 gram copper) until the blue colour disappears.

The antimony is estimated alone on a further quantity of metal, for which 1 gram of filings is treated in a 150-c.c. beaker with 1 gram of potassium iodide, 40 c.c. of HCl, and 40 c.c. water, boiled gently for one hour, filtered, and the precipitate washed with hot dilute HCl (1 : 10), then with hot water, and finally with alcohol. A duplicate filter-paper is treated in the same way, both dried at 100° C. for one hour, and the precipitate weighed as metallic antimony. This, calculated to Sb_2O_4, and subtracted from the mixed tin and antimony oxides, gives the weight of SnO_2, and hence the percentage of tin. W. H. S.

Gutzeit Test for Arsenic. J. A. Goode and F. M. Perkin. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 507-512.)—The authors prefer to use magnesium and ammonium chloride for the reduction in place of zinc and sulphuric acid, as when once started the evolution of hydrogen goes on gently for about two hours without further attention. The apparatus adopted consists of a conical flask, of 250 c.c. capacity, fitted with a dropping funnel, and attached through A to a U-tube containing a 10 per cent. solution of acid cuprous chloride, the exit from the U-tube being restricted by inserting a rubber cork through which passes a glass tube of 5 mm. internal diameter. About 5 grams NH_4Cl are first placed in the flask, then 1 to 2 grams of magnesium turnings, wire or ribbon, added, and 10 c.c. of water run on to the mixture, the apparatus being gently shaken and placed in a basin of cold water. A piece of filter-paper soaked in mercuric chloride solution—or, better, in a strong alcoholic solution of mercuric bromide, which gives a more intense stain—is placed over the outlet and kept there for one hour, or if arsenates are present, for one and a quarter hours, the paper being then placed on a watch-glass, moistened with a few drops of concentrated hydrochloric acid, warmed, the acid poured off, the paper washed with a little water and allowed to dry. Zinc and acid is slightly more sensitive than magnesium with ammonium salts, but with the latter $\frac{1}{1000}$ mgm. of arsenious oxide is easily detectable. The sensibility of the magnesium is diminished by the addition of cadmium salts. W. H. S.



Analysis of Molybdenum Ores. A. Gilbert. (*Zeit. öffentl. Chem.*, 1906, xii., 263-265.)—The following method is applicable to native molybdenum sulphide, and consists in roasting the ore, dissolving the residue in ammonia, filtering, evaporating the filtrate, and igniting the residue obtained. One gram of the finely-powdered ore is placed in a porcelain boat, which is then inserted in a combustion-tube and strongly ignited for three hours. When cold, the contents of the boat are heated for several hours with fairly strong ammonia solution, a little molybdic acid, which sublimes on to the inner surface of the combustion-tube, being also dissolved in ammonia and added to the main bulk. The solution is filtered, the filtrate is evaporated in a platinum basin, and the residue cautiously heated until a constant weight is obtained, care being taken that the bottom of the basin is only heated to a dull redness. The contents of the basin after weighing are dissolved in ammonia, and any insoluble matter subtracted from the total weight. The matter remaining insoluble in the first treatment with ammonia may contain a trace of molybdenum, and this is determined separately by fusing the residue with sodium hydroxide, reducing with zinc and hydrochloric acid, and titrating with standard permanganate solution. W. P. S.

Determination of Potassium by Means of Platinum Tetrachloride in the Presence of Sulphates of the Alkalies and Alkali Earths. K. Regel. (*Chem. Zeit.*, 1906, xxx., 684, 685.)—In the method proposed, the precipitation of the potassium platinum chloride is carried out in the usual way, but without previous separation of lime, sulphuric acid, etc. The precipitate is collected on a filter, washed with alcohol, and then dissolved off the filter with hot water; an excess of magnesium powder is added to the solution, the mixture is warmed, and the precipitated metallic platinum collected on a filter, washed with dilute hydrochloric acid to remove the excess of magnesium, dried, ignited, and weighed. The contamination of the potassium platinum chloride with sodium, calcium, or magnesium sulphate has, therefore, no influence on the determination. As the metallic platinum tends to run through the filter when washed with hydrochloric acid, especially if magnesium and calcium sulphates be present, it is better to wash it, in such case, with dilute nitric acid. W. P. S.

On the Analysis of Sodium Peroxide. Richard Laseker. (*Oester. Chem. Ztg.*, 1906, ix., 164.)—The author has compared the various methods for determining the available oxygen in sodium peroxide, and concludes that Grossmann's modification (*Chem. Ztg.*, 1905, xxix., 138) of Archbutt's method (ANALYST, 1895, xx., 3) is the best. It consists essentially in gradually adding a mixture of 15 c.c. of dilute sulphuric acid (1 : 10) and 2 drops of saturated cobalt nitrate solution to from 0·5 to 0·8 gram of the sample, and measuring the evolved gas over water in some form of jacketed nitrometer.

For a complete analysis, the total alkalinity and the iron and alumina may be determined by the usual methods. The specific gravity may be determined by means of benzene dehydrated by standing over sodium peroxide. Great care should be exercised in the sampling. A. G. L.

APPARATUS.

Improved Extraction Cup. E. B. Warren. (*Chem. News*, 1906, 93, p. 228.)—The essential feature of this cup is that the substance extracted is acted upon by the pure solvent, at the boiling-point of the latter, thus greatly facilitating the extraction of difficultly soluble substances, such as Carnaüba wax by acetone. Two forms are described, that shown in Fig. 1 having a discharge pipe from the bottom, which is carried upwards to a point below the top of the cup, allowing the residue to be dried in a stream of any gas, if liable to undergo change during drying in the ordinary way, while in Fig. 2 the cup containing the substance to be extracted is supported in a tube, and the solvent in the cup kept at a height corresponding to the height of the outer tube. The bottom of either cup is covered by a plug of wool, the material under examination weighed into it, and the cup placed in a flask, into which is poured the solvent. The flask is then attached to a reflux condenser so arranged that the condensed solvent falls back into the cup, percolates through the material, and passes away through the outlet back into the flask.

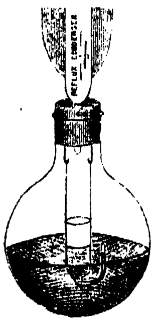


Fig. 1.

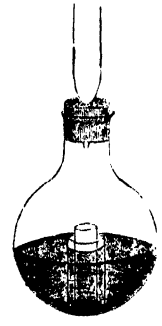
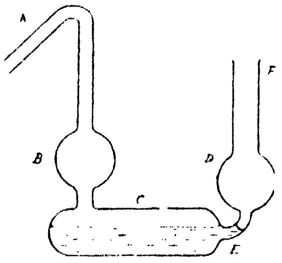


Fig. 2.

W. H. S.

New Form of Absorption Tube. E. P. Perman. (*Chem. News*, 1906, xciii., 213.)—This tube is devised to prevent any possible sucking back of the absorbing liquid into the generating flask during the estimation of ammonia, chlorine (from peroxides), etc.



The long bulb C is 3·5 inches long and 1 inch in diameter; the bulbs B and D 1·25 inches in diameter. The liquid in C should just close the narrow tube at E. A is connected with the generating flask by rubber, cork, or ground-glass joint, and a guard tube, containing glass beads moistened with the absorbent, may be connected to F.

W. H. S.

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REVIEW.

Uric Acid (the Chemistry, Physiology, and Pathology of Uric Acid and the Physiologically Important Purin Bodies, with a Discussion on the Metabolism of Gout). Francis H. McCrudden. (New York: Paul B. Hoeber. Price: paper, $2.50; canvas, $3.00 net.)

The work is a painstaking exposition of all that is at present accurately known as to the conditions of the formation and excretion of uric acid in the animal system. The references given to the literature of the subject are exceedingly copious, and would appear to be exhaustive. The portion devoted to the purines is most instructive, and the whole work should be decidedly valuable to the physiological chemist.

W. J. S.

ANALYSIS AND CONTROL OF MILK-SUPPLIES.

(*From the "Chemist and Druggist" of June* 16, 1906.)

In Section 8*c* of the International Congress of Applied Chemistry held in Rome a very interesting paper was read by A. Lane, of Rotterdam, on this subject. Hitherto the public has been protected from adulteration of milk to a great extent by the systematic taking of samples and assay of "fat" and "other solids," and the supply has in consequence vastly improved in those respects; but there still remains the fact that enormous volumes of milk are supplied for human consumption which come from diseased cows. Tuberculosis is so prevalent that it will probably be demonstrated before long that the mixed supplies of milk from all the counties of England are tainted. Bacteriology fails to detect this except by animal inoculation, which, owing to the time involved and for other reasons, is impracticable. Very welcome, therefore, is any method leading to a ready means of detecting diseased milk. By the ordinary routine of milk analysis it is often possible for grossly diseased milk to pass the standard, and yet it is vastly more important to the consumer that his milk should come from a healthy source than that he should be defrauded of 5 or 10 per cent. of the fat. The chief factor upon which the author depends is what he calls the "catalase number," which is obtained by the introduction of 10 c.c. of the milk and 5 c.c. of hydrogen-peroxide solution into a fermentation-tube, and measuring the gas liberated by the catalase in the milk, which is increased in stale and diseased milk. In his published analyses a most striking contrast is shown between the milk derived from different udders of the same cow suffering from parenchymatous mastitis. The disease was confined to one part of the udder, and the catalase number for that quarter was 6·6, while the healthy part yielded a catalase number 1·2 (healthy fresh milk being unity). The figures in many other diseased conditions are reported, both local in the udder and general. The author has also investigated the effect of milk from diseased cows upon the polarimetric reading, the degree of acidity, and the freezing-point, and his contention is that all these factors should be ascertained in judging a milk. His investigations lead him to the opinion that a lower reading than 4° of the polarimeter, accompanied by a normal or slightly lowered freezing-point, indicates a milk from a diseased cow, and a catalase number above 3 indicates a decomposition advanced to such a degree that the milk should be considered unfit for human consumption.

* * * * *

NARROW ESCAPE OF A LABORATORY STAFF.

A deliberate attempt was made some months since to poison the entire staff of one of the most important of Public Departments, the Government Laboratory at Demerara, and it is clear that but for a succession of fortunate circumstances the design would have succeeded. For some time past a person had been lurking about the laboratory, and on two occasions at least the premises had been illegally entered. On Friday, June 1, a quantity of spirits submitted for analysis was stolen, and although the police at Broad Street Station, a few yards distant from the institution, were instructed to keep a special watch, the thief was not again seen near the laboratory. Just after that date Professor Harrison, the head of the department, was incapacitated for two or three days by a mysterious illness, and there is little doubt that he was then suffering from arsenic poisoning. It would appear that the miscreant again entered the laboratory on the following Wednesday night. A false key was evidently used, nothing being broken or forced at doors or windows. Samples were tampered with, and this fact was immediately reported to the Government on Thursday by Professor Harrison. In the afternoon the Professor, after drinking several glasses of water, was almost overcome with a severe headache. Feeling giddy and unfit for work, he left the office earlier than usual. He was very ill that night. Though far from well the director was able to attend his office on Friday, but after again partaking of the water he had a renewal of his severe illness, and was compelled to return home. On Saturday Professor Harrison was unable to leave his bed, and on the following day his condition was so alarming that medical aid was summoned. The assistants at the laboratory were seized with similar distressing symptoms, though, fortunately, their illness did not take the same serious turn Mr. P. V. Garraway, the second assistant analyst, Mr. Christiani, the director's clerical assistant, Mr. Matthews, the fourth clerical assistant, and the messenger, Tambi, were all affected in more or less degree.

The director was quite unable to leave his house on Monday, and the work of the department was carried on by those of his juniors, who made their appearance at the laboratory every morning—all victims of the same disorder, but never suspecting its origin. While the assistants were discussing the strange circumstances that every member of the staff were affected, it occurred to Mr. Garraway that the trouble might be due to lead-poisoning. The drinking water taken from the filter was subjected to a rough analytical test. Cursory though the examination was, it led to the discovery that arsenic had been placed in the water. Mr. Garraway lost no time in inspecting the filter. At the top was found this substance, in quantity sufficient to poison hundreds of people. The director was communicated with, and realizing the grave import of the news, he ordered that a careful analysis be made of the water. This was done without delay by Mr. E. W. F. English and Mr. Garraway. The water was seen to be heavily charged with arsenic, which, placed in the filter presumably between Wednesday night and Thursday morning, had been slowly but gradually poisoning the drinking supply at the laboratory. Happily arsenic is an extremely insoluble substance. To this fact and to the providential discovery of the poisoned source is due the escape of Professor Harrison and his assistants, who for several days ran exceedingly narrow risks of a terrible death. On further investigation it transpired that the poisoner, with double designs on the Professors, had actually entered that gentleman's office and dropped arsenic into the ice-pitcher kept for the director's own use. Hence the reason why the Professor was taken ill so suddenly, and at an earlier stage than Mr. Garraway and the others. For the first time since its institution, the laboratory had to be closed without notice, the entire staff having been rendered unfit for duty.

Professor Harrison, the shock to whose system has been severe, is invalided home to England.

OCTOBER, 1906. Vol. XXXI., No. 367.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE METHOD OF ANALYSIS OF MILK USED IN THE GOVERNMENT LABORATORY FOR SAMPLES REFERRED UNDER THE SALE OF FOOD AND DRUGS ACTS.

By H. DROOP RICHMOND, F.I.C., and E. H. MILLER.

(*Read at the Meeting*, *July* 14, 1906.)

INTRODUCTION.

It is remarkable that so few investigations into the validity of this method have been made and published. It appears to have been used for more than thirty years, and was first described by Dr. James Bell, the then Principal of the Somerset House Laboratory, in Part II. of his "Analysis and Adulteration of Foods" (see ANALYST, viii., 141). The process may be considered as consisting of two parts, the maceration method of estimating fat and solids-not-fat, and a correction for loss of solids by fermentative change. The maceration method has during the thirty years of use undergone no essential modification, and except in minor points of detail is used to-day as described by Bell. The correction to be made has, however, undergone a radical change. As described by Bell, the correction took the form of a time allowance, and this was severely criticised, notably by Stokes (ANALYST, xii., 226) and Allen (ANALYST, xii., 231, "Commercial Organic Analysis," p. 203), and undoubtedly this correction was liable to grave errors. It has now been superseded by a process of correcting for loss by the estimation of some of the more important products of decomposition (alcohol, volatile acids, and ammonia); the method was published in a report to the Local Government Board (see Report of the Milk Standards Committee, Blue-Book, Cd. 484, 1901, p. 404), and again by Thorpe (Transactions of the Chemical Society, 1905, p. 206). The latter paper contains, in addition to experimental proof that alcohol, volatile acids (acetic and butyric), and ammonia are actually formed in sour milk, a further correction for butyric acid should the amount of this be appreciable.

The only investigation made into the maceration method outside the Government Laboratory of which we are aware was carried out by Bevan and Hehner in conjunction with one of us, and the results are summarized in the Blue-Book cited above (pp. 391 and 392); the directions followed were those of Bell, which are not very explicit, and the method was not carried out in precisely the manner employed at the Government Laboratory. It appeared to us desirable, considering the importance of the method in the administration of the Sale of Food and Drugs Acts, to investigate its accuracy, and with the concurrence, and partly even at the request of Dr. Thorpe, we commenced the present research. By the kindness of Dr. Thorpe, one of us was enabled to receive personal instruction in the method from Mr. C. Simmonds, and we believe that our procedure differs in no essential particular from that employed in the Government Laboratory. We took up the investigation with more interest, as we were aware that the method had been subjected to much criticism based for the most part on no direct experimental data, and it appeared desirable to see how far the criticisms were valid.

The research divided itself into two main heads:

1. The maceration method itself, which again could be subdivided into—
    (*a*) The estimation of fat; and
    (*b*) The estimation of solids-not-fat; and
2. The allowances for decomposition.
    This portion of the investigation may be considered as—
    (*a*) The alcohol correction;
    (*b*) The volatile acid correction;
    (*c*) The ammonia correction, including other corrections for proteid change; and
    (*d*) Corrections not dealt with in the Government Laboratory.

Our general conclusions are that, although we have found several sources of error both in the method and in the corrections, they are comparatively trivial, and hardly affect the validity of the process. On the other hand, we have been enabled by the analysis of the decomposed milk to make a satisfactory estimate of the composition of the original milk, more especially if the modifications of the method and the corrections we propose are employed. At the same time, we must confess that the method requires more personal attention than could easily be devoted to it by public analysts, and for this reason we conceive that adventitious error is more likely to occur.

I. The Maceration Method.

Estimation of Fat in Fresh Milk.—It has been shown by Thorpe (*loc. cit.*), and by Hehner, Bevan and Richmond (*loc. cit.*, and "Dairy Chemistry," p. 94), that the results of estimation of fat by the maceration method agree with those obtained by other methods which are admitted to give substantially accurate results, and we have therefore devoted but little time to this. We have, however, incidentally obtained a few results which confirm the accuracy, and have been favoured by Mr. Simmonds with a few others obtained on samples of homogenized milk.

The results are:

No.	Fat by Maceration Method.	Fat by other Methods.
1.	3·92, 3·93*, 3·94*	3·95
2.	3·44,* 3·42*	3·46
3.	3·81,* 3·84*	3·87
4.	3·88, 3·91	3·92
5.	3·90, 3·83	3·85
6.	3·86, 3·86	3·90

Samples 1, 2, and 3 were homogenized milk, and the results marked * are the work of Mr. Simmonds.

The only remark that it is necessary to make is that when the milk before extraction of the fat is neutralized, using phenolphthalein as indicator, the ethereal solution contains the whole of the phenolphthalein which is weighed with the fat, and from which it can be separated, if desired, by taking advantage of its insolubility in petroleum ether. As, however, the amount of phenolphthalein added need not exceed a milligramme, no appreciable error is caused by neglecting this.

Estimation of Fat in Sour Milk.—In the Blue-Book cited, a number of comparisons of fat in fresh milk and in the milk after it has been allowed to become sour are given, and it is there shown that the fat in the sour milk varies from 0·06 per cent. more to 0·15 per cent. less than in the fresh milk, and averages 0·05 per cent. less.

Thorpe (*loc. cit.*) reproduces most of the results and adds a few more, which show substantially the same agreement. We have examined nineteen samples of milk by this method, and, though our limits are slightly wider than those given above, find that the results on eighteen of the samples of sour milk are in very fair agreement with those obtained when fresh by Gottlieb's method. In one sample, however, which is queried in the table of results, the "fat" in the sour milk is more than 0·5 per cent. high; in this case the milk had undergone a most unusual decomposition, and we were able to prove that the ether extract was by no means all fat. The ethereal solution was quite opalescent, and when the ether was evaporated and the residue taken up with petroleum ether, a considerable residue was left (the weight of this was added to the solids-not-fat), and the petroleum ether was also opalescent. After washing the petroleum ether solution with slightly alkaline water it became clear, but on evaporating and drying the "fat" was turbid, and on again taking up with petroleum ether a flocculent residue was left; a second flocculent residue was obtained on evaporating the clear solution. Owing to an accident, the fat after these treatments could not be weighed, but there is no doubt that the figure given (6·41 per cent.) is too high. We shall refer to this sample again; and the results obtained on it indicate that amino acids resulting from the hydrolysis of the proteids were contained in the ethereal solution.

We are of opinion that some of the difference between the results obtained on the fresh milk and those on the sour milk is due to the difficulty of completely redistributing the fat in the curdled milk. Examination of an apparently well-mixed sample of sour milk with a low-power lens shows the presence of quite large particles of cream, and no amount of whisking with a wire brush appears to reduce the milk to the same homogeneous condition easily obtained with fresh milk. We have had one sample from which the label had been apparently soaked off in warm water and another substituted, in which it was impossible to obtain concordant results for fat.

Eight determinations, by the maceration, Gottlieb, and Werner-Schmid methods, gave the following figures: 4·18, 4·05, 4·03, 4·51, 3·67, 3·73, 4·76, 2·78.

Many samples also on mixing yield a portion of their fat in a churned condition, which adheres to the wire brush; we have in these cases made a separate estimation of the churned fat.

We have been quite unable to extract the fat with eight treatments with ether, as prescribed in the description of the method, and have found twenty to thirty treatments necessary. We have also been unsuccessful in obtaining a residue from which we can obtain no more fat. We have usually stopped extracting when six to eight successive treatments yield only a mgm. or two of fat. This may be due to the fact that we have worked in aluminium basins, instead of the platinum vessels used at the Government Laboratory, and that this metal is too soft to permit of very efficient grinding.

Estimation of Solids-not-fat in Fresh Milk.—We have made a number of comparisons of the solids-not-fat by the maceration method with those obtained by the Society of Public Analysts' method, and find that invariably the former are higher than the latter. Twelve samples gave the following results:

Maceration Method.	S. P. A. Method.	Maceration Method.	S. P. A. Method.
9·14	8·97	8·54	8·28
9·58	9·34	7·96	7·85
9·68	9·40	9·28	9·10
8·37	8·24	9·04	8·74
9·33	9·07	8·91	8·83
9·11	8·86	9·05	8·90

The average difference was 0·20 per cent.

One of us has suggested (Blue-Book, Cd. 484, p. 274) that the difference was due to the presence of milk-sugar in the hydrated form, but this suggestion was combated by Thorpe (*idem.*) and denied by Lewin (*loc. cit.*, p. 350). We have, therefore, made a number of experiments on this point.

(*a*) Weighed quantities of milk-sugar were dissolved in water and evaporated to a firm paste; on treatment with ether this became at once solid, and was finely powdered. After drying to constant weight, the quantity left corresponded neither to the weight of hydrated sugar nor of anhydrous sugar, but to an intermediate weight.

Hydrated Sugar taken.	Residue obtained.	Calculated for Anhydrous Sugar.
1. 0·5120	0·4955	0·4884
2. 0·6630	0·6470	0·6298

(*b*) We repeated Schmöger's experiment (*cf.* Veitch, ANALYST, xi., 141). The solids-not-fat were estimated in 10 grams (*circa*) of milk by the maceration method; in another portion of the same milk of almost identical weight a weighed quantity of milk-sugar was dissolved, and the solids-not-fat estimated in this; the difference between the two weights corresponded neither to the milk-sugar added nor to the anhydrous sugar calculated therefrom.

Hydrated Sugar added.	Residue obtained.	Calculated for Anhydrous Sugar.
1. 0·5000	0·4775	0·4750
2. 0·4245	0·4232	0·4033
3. 0·4349	0·4301	0·4132

A similar experiment in which the total solids by the Society of Public Analysts' method in a sample of milk was subtracted from the total solids of the same quantity of milk to which a known weight of milk-sugar was added yielded an amount that differed from the weight calculated for anhydrous sugar by less than a mgm.

(*c*) As we have found that when hydrated milk-sugar is stirred with water (1 gram to 10 c.c.) it produces a fall of temperature of 0·55°, while anhydrous milk-sugar gives a rise of temperature of at least equal extent, we endeavoured to use this as a means of investigating the question. With ordinary milks the change of temperature was slight, but the solids-not-fat of milk obtained by repeatedly extracting and finely grinding the total solids obtained by the Society of Public Analysts' method with ether tended to give about 0·1° higher temperature than the solids-not-fat obtained by the maceration method. When the temperature change was exaggerated by using the solids of the milk to which milk-sugar had been added there was a marked difference. Samples 2 and 3 of solids-not-fat of milk to which milk-sugar had been added, obtained by the maceration method, caused a fall of temperature of 0·3°, while the solids-not-fat obtained by extracting the solids by the Society of Public Analysts' method of the milk to which milk-sugar had been added gave a rise of 0·3°.

We think, therefore, that there is no doubt that the solids-not-fat obtained by the maceration method contain a portion of the sugar as hydrated sugar, but the amount of water of hydration, which we estimate averages about 0·1 per cent., is not sufficient to explain the difference between the results.

Another source of error in the maceration method is due to the presence of aldehydes in the ether. It is well known that ether contains aldehyde, and that when this is removed, the ether, after a short interval, again contains aldehyde. We have found that ether free from aldehyde will give a marked Schiff reaction if shaken for a few minutes in daylight. We also find that milk solids remove the aldehyde completely from ether, and this appears to be due to a condensation of the $-COH$ group with the free amino groups of the proteids. The solids-not-fat obtained by the maceration method are always more acid than the milk, and the aldehyde figure is less, the increase of acid and the decrease in the aldehyde figure being within the limits of experimental error identical. These figures afford data for the estimation of the increase of weight due to the condensation of the aldehyde, and assuming that it is acetaldehyde, the error is almost constantly 0·03 per cent. unless freshly-distilled aldehyde-free ether be used. The solids-not-fat by the maceration method also give a faint Liebermann reaction pointing to the condensation of glyoxylic acid, but this cannot be large, as, if it were, the increase of acid would be larger than the decrease of the aldehyde figure.

Even this addition does not explain the whole of the difference between the maceration and Society of Public Analysts' methods, and the marked browning of the residue in the latter method suggests that the remainder of the difference is due to the results obtained by it being too low; this conclusion is strengthened by the fact that evaporation over a large surface, whereby browning of the residue is avoided, gives slightly higher results.

In sour milks the error of the maceration method due to water of hydration will be reduced, as the amount of milk-sugar is less than in fresh milk; but, on the other hand, a portion of the milk-sugar may be converted into hexoses, which will have the same effect on the weight as hydration.

The exact details of the method we have employed are these: About 10 grams of milk are weighed into a basin of aluminium about 3 inches in diameter and a little over 1 inch high, with a flat bottom, provided with a flat-ended glass stirrer. Two drops of a 0·5 per cent. solution of phenolphthalein are added, and approximately $\frac{N}{10}$ strontia solution run in till a faint pink colour appears. The contents are evaporated to a damp paste on the water-bath, when the basin is transferred to a hot plate, and the paste mixed with the stirrer; at a certain point in the evaporation the paste comes away from the basin, and by careful manipulation both basin and stirrer can be obtained practically clean. On further evaporation and stirring, the paste begins to get into a condition in which it can be broken up and rubbed into pieces, and at this stage it is removed from the hot plate and about 20 c.c. of ether are added. On gentle rubbing with the stirrer the solids begin to go to pieces; the stirrer and basin are now scraped with a knife or spatula to bring any small portions of solids adhering to the sides under the ether, and the solids are gently rubbed to a powder. The ether is decanted through a weighed filter 9 cm. in diameter, and the solids again treated with ether. The solids at this stage are in a condition in which they can be ground up to a fine powder; the ethereal solution is allowed to settle, and the ether decanted through the filter; without any further addition of ether the solids are now ground to a very fine powder. We prefer to do this with only a very little ether in the basin, as it is then easy to see the larger portions which can be ground up one by one. A further addition of ether is made, and the solids further ground; the ether at this stage looks like whitewash, and the solids take some minutes to subside sufficiently to allow of decantation. After about six or eight treatments in this manner the solids are allowed to air-dry, the portions clinging to the stirrer and sides of the basin scraped down, and 5 c.c. of alcohol and a few drops of water are added; the solids are well mixed with the alcohol, and the basin is placed on the hot plate and evaporated till the paste begins to go to pieces, when the solids are again treated as before. A second treatment with alcohol and a further six to eight extractions with ether are given; the filter gradually becomes partially blocked with the finely-divided solids, but never to such an extent that filtration stops. The solids are air-dried, and then dried in the water-oven to constant weight. We have not usually weighed the solids-not-fat in a weighing-bottle; although they are hygroscopic, we have satisfied ourselves that no appreciable error is due to this cause. The final weight is known from previous weighings to within a very small amount, and consequently the time of weighing is very short, and we have proved that not more than a few tenths of a mgm. of hygroscopic moisture are taken up during the weighing. The top of the filter where fat collects is cut off and cut into pieces and thoroughly washed with ether; the knife or spatula is wiped on some of the pieces cut off to remove any particles of solids, and the filter containing the pieces cut off is dried in the water-oven to constant weight. The ether is distilled, and the residue of fat weighed; the fat is extracted with petroleum ether, and the small residue insoluble in this solvent subtracted from the weight; this usually consists of phenolphthalein, and its weight may be neglected without appreciable error.

The ether employed by us has been methylated ether (specific gravity 0·720) dried with calcium chloride.

II. The Allowances for Decomposition.

The Alcohol Correction.—We have usually taken about 75 grams for the estimation of alcohol. The quantity is weighed into a 300-c.c. flask, and half neutralized with $\frac{N}{2}$ soda solution; about half the volume is distilled and collected in a small flask, and neutralized with $\frac{N}{2}$ soda, using litmus-paper as indicator. From this 25 c.c. is distilled, and the density taken at 60° F. by a Sprengel tube. From the density the percentage of alcohol is calculated by the formula given in the Blue-Book, and from this the correction is calculated.

We believe that the alcohol correction is very near the truth.

The Volatile Acid Correction.—We have in every sample estimated the volatile acids by the method given in the Blue Book, but do not think that the results by this method at all represent the amount of volatile acid, except when the quantity is very small. The method is, in our opinion, subject to several errors, and is liable to be quite 50 per cent. from the truth. In the first place, carbonic acid is always present, and is driven off on evaporating the half-neutralized milk to dryness, and as this is partially estimated, using phenolphthalein as indicator, as acetic acid, an error is thereby produced. The error is a double one, as not only is a correction made for carbon dioxide lost during the production of acetic acid, but the carbonic acid itself is retained by the strontia, and is weighed with the solids. The sum of these two errors we estimate as about 0·05 per cent. Against this we find that the whole of the volatile acids are not driven off on evaporation to dryness, and the error due to this cause will partly compensate the error due to carbonic acid. There is another source of error, due to a possible volatility of lactic acid, and to the conversion of a portion of this acid into a lactone. Against this there is the formation of amino acids by hydrolysis of the proteids, which would have a feeble acidity to phenolphthalein. We always find that fresh milk on half neutralization and evaporation gives a very slight increase of acidity; and in one sample of sour milk we found that there was an increase of acidity on evaporating the half-neutralized milk, and this result was confirmed on repetition. Probably when the volatile acidity lies between 0·1 and 0·2, as in the majority of our samples, the method gives approximately correct results; in other cases it is quite erroneous.

The following experiments will show this:

0·0162 gram acetic acid added to milk ...	found 0·0084 gram.
„ „ „ „ „ „ ...	„ 0·0092 „
0·0162 gram acetic acid + 0·07 gram lactic acid added to milk	„ 0·0165 „
0·0125 gram acetic acid + 0·0414 gram lactic acid added to milk + the solution saturated with carbonic acid (contained 0·0031 gram CO_2)	„ 0·0192 „

In the presence of much volatile acid, we have added to the milk from which the alcohol had been distilled a quantity of acid exactly equal to the soda used for half neutralizing, and have distilled this down to a small bulk, and then added water in successive quantities of about 25 c.c., and distilled this off till the distillate was practically neutral. Thorpe (*loc. cit.*) has given practically this method, but apparently has not added any acid; as he directs that the volatile acids may be estimated in the distillate by any convenient method, we employed that of Duclaux, which appears to us not only to be capable of considerable accuracy, but which enables us to estimate the relative proportions of even three acids in a mixture, and to indicate, if the modification we have devised be employed, what the acids present are. We have found it essential to add sufficient acid to neutralize the soda added, as in some cases the volatile acid exceeds half the total acidity.

Our mode of working is this: The mixed distillates neutralized to phenolphthalein are made up to 250 c.c., and an aliquot portion taken for fractional distillation; to this is added a quantity of N sulphuric acid, slightly greater than is necessary to neutralize the soda used. Eight successive fractions, each about one-ninth of the total volume, are distilled, and separately titrated with $\frac{N}{10}$ strontia; 25 c.c. of water is added to the residue, and a further 25 c.c. distilled and titrated, and, if desired, this treatment may be repeated. From the last titration the total quantity of volatile acid is obtained by extrapolation of the results; the total quantity is always slightly less than the total acidity of the first distillate, due no doubt to the presence of a little lactic acid. As much as possible of the first distillate is made up to a convenient bulk, after adding a quantity of N sulphuric acid in slight excess of that required to neutralize the soda, and exactly one-third is distilled; this is made up to a convenient bulk, and exactly one-third is again distilled. The last distillate is made up to 100 c.c., and eight fractions of about 11 c.c. each are distilled and separately titrated. The residue in the flask and condenser is washed out and titrated to obtain the total quantity.

For the calculation of the results we have slightly modified the formulæ given by one of us (ANALYST, xx., 193) to:

$y = x^{2 \cdot 1}$ for butyric acid;
$y = x^{1 \cdot 18}$ for propionic acid; and
$y = x^{\frac{2}{3}}$ for acetic acid.
y = quantity of acid left in retort; total quantity = 1.
x = volume of liquid left in retort; total quantity = 1.

For each fraction we calculate the proportion of each of the acids which would distil, and calculate the ratios of butyric to acetic, and butyric to propionic acid, which correspond to the proportion actually distilled. The ratios from first and last fractions are liable to be slightly erroneous, the first because a small amount of a very volatile acid, as carbonic, would appear in this fraction, and the last because experimental error is greatly magnified; but we usually find that the other six ratios are practically constant for either butyric and acetic acids, or butyric and propionic acids.

The distillation of the third of a third is undertaken in order to decide definitely the composition of the mixture. From the formulæ above, the proportions which would distil, when one-third of one-third is collected, are:

For butyric acid	0·3285
For propionic acid	0·1445
For acetic acid	0·061

Thus, a mixture of butyric and propionic acids in equal proportions should yield approximately a 2 : 1 mixture in the distillate, while a mixture of butyric and acetic in equal proportions would yield a 5 : 1 mixture. It is even possible to deduce the relative proportions of a mixture of the three acids.

We give below the results of three experiments. In the first we have concluded that the acids present were acetic and butyric; in the second, propionic and butyric; and in the third a mixture of the three.

No. 1.—ACETIC AND BUTYRIC ACIDS.

Distillation of Original Distillate.					*Distillation of ⅓ of ⅓.*				
Volume Distilled.	Acid Distilled.	Calculated for—		Ratio.	Volume Distilled.	Acid Distilled.	Calculated for—		Ratio.
		Butyric.	Acetic.	Butyric / Total.			Butyric.	Acetic.	Butyric / Total.
0·108	0·141	0·213	0·073	0·49	0·113	0·190	0·2225	0·077	0·78
0·221	0·2275	0·406	0·153	0·49	0·214	0·343	0·397	0·148	0·78
0·335	0·403	0·5755	0·238	0·49	0·328	0·490	0·566	0·2325	0·77
0·440	0·515	0·708	0·324	0·495	0·437	0·619	0·701	0·318	0·785
0·559	0·6175	0·821	0·421	0·49	0·547	0·726	0·8105	0·410	0·79
0·669	0·711	0·902	0·521	0·50	0·659	0·819	0·898	0·512	0·795
0·779	0·793	0·958	0·6345	0·49	0·773	0·892	0·956	0·628	0·805
0·887	0·8705	0·9915	0·780	0·48	0·891	0·950	0·9905	0·782	0·805

The quantity distilled in ⅓ of ⅓ was 0·193.

The figure calculated for a mixture of 0·495 molecules of butyric to 0·505 molecules of acetic was 0·192.

The ratio of butyric acid in the second distillate should be 0·83.

No. 2.—PROPIONIC AND BUTYRIC ACIDS.

Distillation of Original Distillate.					*Distillation of ⅓ of ⅓.*				
Volume Distilled.	Acid Distilled.	Calculated for—		Ratio.	Volume Distilled.	Acid Distilled.	Calculated for—		Ratio.
		Butyric.	Propionic.	Butyric / Total.			Butyric.	Propionic.	Butyric / Total.
0·104	0·1685	0·206	0·1215	0·56	0·110	0·183	0·217	0·128	0·62
0·215	0·328	0·398	0·248	0·53	0·223	0·357	0·411	0·258	0·65
0·331	0·478	0·570	0·378	0·505	0·333	0·509	0·573	0·380	0·67
0·444	0·609	0·708	0·499	0·53	0·448	0·641	0·713	0·504	0·66
0·558	0·723	0·820	0·6185	0·52	0·554	0·750	0·8165	0·614	0·67
0·656	0·807	0·894	0·716	0·51	0·664	0·839	0·899	0·724	0·66
0·767	0·886	0·953	0·821	0·49	0·775	0·911	0·9565	0·828	0·65
0·875	0·949	0·987	0·914	0·48	0·889	0·964	0·990	0·925	0·60

The quantity distilled in $\frac{1}{3}$ of $\frac{1}{3}$ was 0·242.

The figure calculated for a mixture of 0·514 molecules of butyric to 0·486 molecules of propionic was 0·239.

The ratio of butyric acid in the second distillate should be 0·70.

No. 3.—ACETIC, PROPIONIC, AND BUTYRIC ACIDS.

Distillation of Original Distillate.

Volume Distilled.	Acid Distilled.	Calculated for—			Ratio.		
		Butyric.	Proprionic.	Acetic.	$\frac{\text{Butyric}}{\text{B + Ac.}}$	$\frac{\text{Butyric}}{\text{B + P.}}$	$\frac{\text{Butyric}}{\text{B + 2P + A.}}$
0·105	0·169	0·208	0·1225	0·071	0·715	0·545	0·62
0·214	0·324	0·397	0·2475	0·148	0·71	0·51	0·60
0·326	0·467	0·563	0·372	0·231	0·71	0·50	0·60
0·432	0·588	0·695	0·487	0·314	0·72	0·49	0·60
0·547	0·705	0·8105	0·607	0·410	0·735	0·48	0·62
0·661	0·8105	0·897	0·721	0·514	0·75	0·46	0·61
0·772	0·897	0·955	0·825	0·627	0·78	0·44	0·63
0·895	0·950	0·991	0·930	0·7775	0·81	0·33	0·63

It is seen that, the ratios calculated on the supposition that the acids consist of acetic and butyric and propionic and butyric, neither of them give constant figures, but when calculated for a mixture of 2 molecules propionic and 1 acetic the agreement is satisfactory. On this assumption the total quantity distilled in $\frac{1}{3}$ of $\frac{1}{3}$ should be 0·248 of the total. We actually found 0·251, and the ratio of propionic to acetic in the last distillate should be 5 to 1.

Distillation of $\frac{1}{3}$ of $\frac{1}{3}$.

Volume Distilled.	Acid Distilled.	Calculated for—			Ratio.		
		Butyric.	Propionic.	Acetic.	$\frac{\text{Butyric}}{\text{B + Ac.}}$	$\frac{\text{Butyric}}{\text{B + P.}}$	$\frac{\text{Butyric}}{\text{B + 5P + A.}}$
0·106	0·190	0·210	0·124	0·072	0·85	0·77	0·79
0·217	0·360	0·4015	0·2505	0·1505	0·835	0·725	0·75
0·333	0·521	0·573	0·380	0·247	0·84	0·73	0·76
0·445	0·652	0·707	0·501	0·325	0·83	0·73	0·765
0·554	0·761	0·8165	0·614	0·416	0·86	0·725	0·765
0·667	0·850	0·9025	0·730	0·5225	0·86	0·695	0·76
0·781	0·921	0·959	0·8335	0·637	0·88	0·70	0·76
0·894	0·969	0·991	0·929	0·776	0·90	0·64	0·74

The ratio of butyric acid in the second distillate should be 0·78.

It is noticed in every case that the ratio of butyric acid in the distillate of $\frac{1}{3}$ of $\frac{1}{3}$

is slightly less than that calculated, and this is partly due to the fact that there is always a slight loss during the two distillations, and as butyric acid is the most volatile of the compounds present, the loss will be proportionately larger of this acid than of the others.

We think that we may claim that the above figures show that a fairly accurate estimation of, and a determination of the nature of, the acids present is possible by Duclaux's method as modified.

We append a table showing the total volatile acids expressed as c.c. of N alkali per 100 grams of milk estimated by the method described in the Blue Book and Duclaux's methods respectively in five samples which have been high in volatile acids :

No.	Blue Book Method.	Duclaux's Method.	Ratio.
7.	7·35	12·6	1·71
9.	5·7	9·75	1·71
11.	7·68	12·2	1·59
13.	14·0	21·6	1·54
15.	12·35	20·7	1·68

As to the corrections to be made for volatile acids there is some doubt. When the amount has been small we have assumed them to be acetic acid alone, and have applied the correction given in the Blue Book—*i.e.*, an addition of 25·5 parts for each 60 parts of acetic acid. This correction we prefer to that given by Thorpe—*i.e.*, 62 parts for each 60 parts of acetic acid—as the latter correction assumes that acetic acid was produced by oxidation, and the oxygen was part of the original milk.

For butyric acid Thorpe gives a correction of 92 parts for each 88 parts of butyric acid, but this correction assumes that the weight of lactic acid from which it was supposedly formed was equal to the weight of the milk-sugar giving rise to it. This is the case if the milk-sugar is supposed to exist in the solids as hydrated sugar, but we have shown that this is only partially the case, and prefer to calculate the loss to anhydrous sugar, and modify the correction to 87·5 parts for each 88 parts of butyric acid.

We are, however, by no means satisfied that this correction is justified; it assumes that the butyric acid is derived from lactic acid. It is well known that the organisms producing lactic acid do not form more than about 1 per cent. of lactic acid, as their action is inhibited by this amount of acid. In some of our samples the total acidity has been equal to nearly 3 per cent. of lactic acid, and as the greater portion of this has been butyric acid, it follows that it cannot have been derived from lactic acid.

We assume that in every case of fermentation the milk-sugar is hydrolysed to glucose and galactose, and we therefore take a hexose as our starting-point. To explain the formation of butyric and propionic acids, we assume the following reaction to take place :

$$3C_6H_{12}O_6 = 2CH_3CH_2CH_2COOH + 2CH_3CH_2COOH + 4CO_2 + H_2 + 2OH_2,$$

which gives a total loss of 94·5 for 88 parts of butyric and 74 parts of propionic acids. This is the proportion in which we have found these acids, and we have therefore used this correction.

For butyric and acetic acids there are so many equations which can be drawn out to explain their formation, each of which gives a different correction, that we have not attempted to do this, but have used the corrections given above. That they are not quite correct is shown by the agreement of these samples not being quite so good as the others.

The Ammonia Correction.—We are very doubtful as to the validity of this correction. Though during the hydrolysis of the proteid a portion of the carbon and hydrogen may be converted into volatile non-acid compounds, we do not think that the whole of the nitrogen is converted into ammonia with total loss of the other constituents. We are also doubtful whether the colour produced by Nessler solution is all due to ammonia, as it is sometimes so markedly different in tint from that produced by the standard solution of ammonium chloride. As the whole correction is usually small we have accepted it, as we have found that there are changes in the proteids on evaporation of the milk which no doubt involve a slight loss.

We have in many samples estimated the aldehyde figure (*cf.*, Steinegger, ANALYST, xxxi., 46, and ourselves, ANALYST, xxxi., 224), both in the sour milk and in the solution of the solids obtained by evaporating the half-neutralized milk.

The table below gives our results:

No.	Aldehyde Figure in Fresh Milk.	Aldehyde Figure in Sour Milk.	Aldehyde Figure after Evaporation.	NH_3, Correction.
6.	19·4°	24·0°	17·5°	0·01
7.	19·4°	24·1°	15·5°	0·02
8.	19·4°	24·1°	16·9°	
9.	19·4°	21·3°	15·1°	
10.	19·4°	27·6°	16·3°	0·02
11.	19·5°	29·8°	13·9°	0·06
12.	19·4°	27·5°	19·4°	0·01
13.	19·4°	35·6°	14·9°	0·09
14.	19·4°	32·0°	16·3°	0·07
15.	19·6°	22·7°	16·7°	0·01
16.	19·4°	26·7°	15·7°	0·02
17.	14·4°	23·1°	10·9°	0·03
18.	19·4°	32·9°	18·2°	0·12
19.	14·5°	27·1°	11·8°	0·10

These figures show that in all the samples of sour milk hydrolysis has taken place with the formation of amino acids, but that on evaporating, these amino acids condense either to form di-keto-piperazine derivatives or polypeptids; the hydrolysis would cause an increase in weight, but the condensation a loss, and as the figure is always lower after evaporation than in the fresh milk, this indicates that a slight additive correction should be made. It is noticed that when there is much ammonia developed there is also a high aldehyde figure.

We have examined one sample (No. 5) which is not included in the above list, which gave an aldehyde figure after evaporation of over 300°; on evaporation it smelt like glue, and became a plastic mass. This is the sample referred to as yielding too high an ether extract. The distillate, which should have contained only alcohol, was found to be strongly alkaline with ammonia or an amine, and the acidity was higher than it could have been had all the milk-sugar originally present been converted into lactic acid. The solids-not-fat were higher in the sour milk than in the fresh milk, and the system of corrections adopted at the Government Laboratory entirely failed to give correct results. This sample had undergone a most unusual form of decomposition, but as it was not originally a normal milk we do not consider that it invalidates the system. As the unusual decomposition, which was not realized till the sample had been used up, prevented an accurate analysis being made, we cannot speak very definitely, but there are distinct indications that the further corrections which we propose would have given good results even with this sample.

Corrections not dealt with in the Government Laboratory.—These corrections are three in number; the first is for the lactic acid produced. The usual equation for this is—

$$C_{12}H_{22}O_{11} + OH_2 = 4CH_3CHOHCOOH,$$

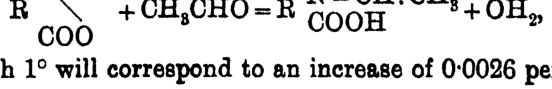
which indicates that 342 parts of milk-sugar produce 360 parts of lactic acid. We have therefore subtracted 5 per cent. of the lactic acid from the weight of the solids-not-fat. We calculate the total acidity, less the volatile acidity plus 0·2 per cent. (to represent the acidity of the milk when fresh), as lactic acid.

The second correction is for the aldehyde taken up from the ether used. We find that the solids-not-fat, which should of course be neutral, are always acid after extraction with ether, and the aldehyde figure has gone down. That this is due to condensation of aldehyde is shown by the substantial agreement of the increase of acidity with the decrease in the aldehyde figure. As the aldehyde figure is affected by the combined error of four titrations, the agreement is good.

No.	Increase of Acidity.	Decrease of Aldehyde Figure.
	7·4°	7·4°
6.	10·5°	9·1°
8.	7·6°	9·8°
10.	8·2°	6·7°
12.	4·3°	5·1°
14.	9·9°	10·0°
16.	8·2°	8·0°
17.	5·6°	5·5°
18.	5·6°	4·7°
19.	5·8°	5·5°

All figures are expressed as c.c. of $\frac{N}{10}$ alkali per 100 grams.

Assuming that the following equation takes place:



$$R\overset{NH_2H}{\underset{COO}{\diagdown}} + CH_3CHO = R\begin{matrix}N=CH.CH_3\\COOH\end{matrix} + OH_2,$$

it is seen that each 1° will correspond to an increase of 0·0026 per cent. of weight.

When the sour milk contains a large amount of butyric acid the increase of acidity does not agree with the decrease of the aldehyde figure. In these samples an odour of butyric acid is noticed on extracting and drying, and the solids-not-fat are either neutral or alkaline. In this case we calculate what the increase of acidity should be from the aldehyde figure, and take the difference between this figure and the figure actually found to represent loss of butyric acid by multiplying each degree by 0·0088.

In cases where the aldehyde figure of the evaporated milk is higher than 20° we should make a correction to be subtracted from the solids-not-fat for the weight of water taken up on hydrolysis. The equation of this change is:

$$R-CO-NH-R^1+OH_2=R-COOH+NH_2R^1,$$

and each degree of aldehyde figure above 20° would represent a gain of 0·0018 per cent. of water. The abnormal sample previously referred to is the only one we have examined which would be subjected to this correction.

Discussion of Results.—In the table on p. 331 we give the analytical figures for nineteen samples of milk, which were examined when fresh by the Society of Public Analysts' method and when sour by the method used in the Government Laboratory.

Taking the whole of the samples, it is seen that the fat estimated by the maceration method in the sour milk varies from 0·10 above to 0·18 below the fat estimated by the Society of Public Analysts' method in the fresh milk, and averages 0·03 per cent. less. The solids-not-fat of the whole of the samples vary, when corrected by the methods used in the Government Laboratory, from 0·28 above the solids-not-fat in the fresh milk determined by the Society of Public Analysts' method to 0·27 per cent. below; while if the corrections that we propose are used in addition the variation is from 0·32 above to 0·20 below; the differences average +0·08 per cent. and +0·06 per cent. respectively. For milks that have undergone the normal decomposition—*i.e.*, in which chiefly lactic acid is produced—the figures are:

	Maximum.	Minimum.	Average.
Government Laboratory	0·28	−0·11	+0·14
R. and M.	0·17	−0·19	+0·07

Even when there is a marked alcoholic fermentation or a strong butyric fermentation the agreement is still fair, and does not exceed the limit laid down by Thorpe (0·3 per cent.) (*loc. cit.*) as being sufficiently satisfactory.

We have included two samples which contained about 25 per cent. of added water, and here the agreement is satisfactory. These samples were made up by mixing known weights of the milk immediately preceding them in the table and of water, and the amount of water calculated from the analysis of the sour milk after correction corresponded to within a few tenths of 1 per cent. with that actually added.

As showing what different fermentations milk may undergo, we may mention that Nos. 8 and 9 and Nos. 10 and 13 were each taken from the same bulk of milk, though at slightly different times, yet the character of the decomposition varied widely.

It is of interest to mention that in the two samples in which butyric fermentation had taken place, and which give results for solids-not-fat on correction rather

No.	Fat (Original).	Fat (Sour).	Difference.	Solids-not-fat (Original).	Solids-not-fat (Sour).	Alcohol.	Correction.	Butyric Acid.	Propionic Acid.	Acetic Acid.	Volatile Acid (Correction).	Ammonia.	Correction.	Aldehyde Correction.	Loss of Butyric Acid.	Lactic Acid.	Correction.	Solids-not-fat, Government Laboratory.	Solids-not-fat, R. and M.	Age.
																				Days.
1.	2·77	2·78	+0·01	8·29	8·00	0·165	0·32	—	—	—	—	0·014	0·08	−0·03	—	1·38	−0·07	8·40	8·30	246
2.	3·66	3·62	−0·04	8·95	8·90	0·021	0·04	—	—	0·136	0·06	0·009	0·05	−0·03	—	0·94	−0·05	9·05	8·97	31
3.	3·66	3·53	−0·13	8·95	9·03	0·021	0·04	—	—	0·133	0·06	0·009	0·05	−0·03	—	0·88	−0·05	9·18	9·10	31
4.	4·03	4·12	+0·09	8·83	8·68	0·050	0·10	—	—	0·170	0·07	0·009	0·05	−0·03	—	0·83	−0·04	8·90	8·83	46
5.	5·82	6·41?	?	9·71	9·71?	0·155?	0·30?	—	—	0·231	0·10	0·040	0·21	−0·03	—	2·52?	−0·13?	?	?	266
6.	3·72	3·73	+0·01	8·77	8·85	0·071	0·14	—	—	0·116	0·05	0·002	0·01	0·03	—	1·16	−0·06	9·05	8·96	49
7.	4·06	4·02	−0·04	8·86	7·14	0·271	0·53	0·820	—	0·187	0·90	0·004	0·02	−0·03	0·11	0·25	−0·01	8·59	8·66	49
8.	3·92 M 3·89	4·02	+0·10 +0·13	8·74 9·04	8·72	0·082	0·16	—	—	0·171	0·07	0·001	—	−0·03	—	1·23	−0·06	8·95	8·86	37
9.	3·97	3·81	−0·16	8·77	7·74	0·180	0·35	0·385	0·306	—	0·45	trace	—	−0·03	0·10	0·37	−0·02	8·54	8·59	37
10.	3·84	3·92	+0·08	8·82	8·33	0·366	0·71	—	—	0·078	0·03	0·004	0·02	−0·02	—	0·93	−0·05	9·09	9·02	34
11.	3·90 M 3·86	3·88	−0·02 +0·02	8·90 9·05	7·69	0·092	0·18	0·658	0·236	0·095	0·75	0·012	0·06	−0·02	0·06	0·29	−0·01	8·68	8·71	31
12.	3·61	3·64	+0·03	8·84	8·76	0·075	0·15	—	—	0·138	0·06	0·003	0·01	−0·01	—	0·95	−0·05	8·98	8·92	57
13.	3·82	3·90	+0·08	8·88	7·21	0·258	0·50	1·086	—	0·605	1·34	0·016	0·09	0·01	0·10	0·66	−0·03	9·14	9·20	43
14.	3·71	3·53	−0·18	8·79	8·51	0·016	0·03	—	—	0·162	0·07	0·013	0·07	−0·03	—	1·01	−0·05	8·68	8·60	55
15.	3·48	3·36	−0·12	8·81	7·55	0·015	0·03	0·946	—	0·658	1·22	0·002	0·01	−0·03	0·11	0·53	−0·03	8·81	8·86	40
16.	3·57	3·44	−0·13	8·84	8·81	0·032	0·06	—	—	0·108	0·05	0·004	0·02	−0·02	—	0·91	−0·05	8·94	8·87	36
17.	2·63	2·54	−0·09	6·50	6·49	0·030	0·06	—	—	0·108	0·05	0·006	0·03	−0·01	—	0·88	−0·04	6·63	6·58	36
18.	3·63	3·57	−0·06	8·71	8·74	0·031	0·06	—	—	0·114	0·05	0·024	0·12	−0·01	—	0·97	−0·05	8·97	8·91	33
19.	2·70	2·69	−0·01	6·47	6·35	0·086	0·17	—	—	0·084	0·04	0·019	0·10	−0·01	—	0·90	−0·05	6·66	6·60	33

Nos. 2 and 3 were from the same bulk of milk and taken at the same time.
,, 8 and 9 ,, ,, ,, ,, at different times.
,, 10 and 13 ,, ,, ,, ,, ,,
,, 17 was a mixture of 73·6 parts of 16 and 26·4 parts of water. Water calculated from sour milk solids-not-fat 25·7 per cent.
,, 19 ,, 74·3 ,, 18 and 25·7 ,, ,, ,, ,, ,, ,, 25·9 ,,

higher than the original, the ratio of butyric to acetic acid was 1 : 1, while in those which are low the ratio was 3 : 1. This may be a coincidence, and it is evident that many more samples must be examined before the proper corrections can be established.

We have also examined some solutions of milk-sugar to which a small quantity of sour milk was added to supply the organisms necessary for fermentative change.

The milk-sugar solution used contained 4·77 per cent. total solids, calculated from the amounts of milk-sugar and milk taken, and calculating the sugar as anhydrous sugar, and 4·74 per cent. by evaporation to dryness and drying to constant weight.

The following are the analytical figures:

Age.	Total Solids.	Alcohol Correction.	Volatile Acid Correction.	Total Acidity as Lactic Acid.
12 days ...	4·56	0·35	0·03	0·20
37 days ...	4·84	0·20	0·03	0·20
70 days ...	4·70	0·15	0·04	0·29
85 days ...	4·17	0·78	0·09	0·51

It is evident that the samples had undergone but little change; the volatile acid, which was found to be almost wholly carbonic acid, accounted for about half the total acidity. It is seen that, even making a minus correction for the carbonic acid, the results are still too high, and these experiments afford further proof that in the maceration method a portion of the milk-sugar is weighed as hydrated sugar.

In conclusion, we think we may claim to have shown that—

1. The maceration method for fresh milks gives results slightly too high for solids-not-fat, and about to the same extent that the Society of Public Analysts' method is low.

2. By the method used in the Government laboratory, a satisfactory determination of the composition of the orginal milk can be made, the results, except in cases of high butyric fermentation and other abnormal decompositions, not being more than 0·2 per cent. from the truth.

3. The method for the estimation of volatile acids used at the Government Laboratory is not a good one, and requires modification if any appreciable amount of these acids be present.

4. Certain small additional corrections may be made with advantage.

Considering that in the course of an examination of nineteen samples of milk we have found at least two forms of decomposition (a proteolytic and a butyric-propionic fermentation) which require special corrections, it is evident that many more experiments should be made before the analysis of sour milk can be considered to be on a satisfactory basis.

We would emphasize the fact that our comparatively large proportion of samples showing the butyric fermentation is partly due to a selection of these samples in preference to others, and partly to the special precautions taken to prevent the bottles from bursting. With the normal conditions under which reference samples are kept, we doubt whether more than one of the five would have survived to reach the Government Laboratory.

Finally, we would express our thanks to Dr. Thorpe and Mr. Simmonds, of the Government Laboratory, for the kind way in which they have assisted us in this investigation.

DISCUSSION.

The PRESIDENT (Mr. Bevan), in inviting discussion, said that his own experience was that the fat determinations made in the Government Laboratory in samples of sour milk were generally lower than those obtained by the public analyst on the fresh milk. Mr. Richmond, some seven or eight years ago, had considered the maceration process to be unsatisfactory in the case of sour milk, but had now apparently changed his view. In "Commercial Organic Analysis," Mr. Allen had said, in referring to the Government method: "Some points strongly invite criticism, but the method must be welcomed as an ingenious attempt to effect a satisfactory solution of a very difficult problem." And it seemed now to be generally agreed that the method was an ingenious and fairly satisfactory one.

MR. F. J. LLOYD said that he had done a good deal of work on the examination of sour milk, and should be interested to see what light these improved methods would throw on the changes which took place in such samples. Those changes, it might be mentioned, were never constant. He had found that in genuine milk changes occurred which depended entirely on the nature of the bacteria present; and when a sample was adulterated with water, other changes were brought about by the organisms peculiar to water. He had studied the action of various micro-organisms on sterilized milk, and had found that, while it was fairly easy to obtain accurate results with samples which had only been acted on by the lactic acid bacillus, it was very difficult, when gas-producing organisms (such as *Bacillus coli communis*) were present in large quantities, to obtain all the products of the decomposition and calculate the composition of the original milk. In the majority of cases he had found that the loss was proportional to the amount of acid produced, being almost always half the total acidity calculated as lactic acid. The supposition that what took place in the conversion of milk-sugar into lactic acid was merely an addition of the elements of water to the milk-sugar, and a splitting up into exactly four parts of acid, was incorrect. As a matter of fact, only about 60 per cent. of the milk-sugar was actually converted into lactic acid, other compounds being formed from the remainder.

Mr. FAIRLEY said that he could endorse what Mr. Lloyd had said as to the varying effects produced in milk by different bacteria. Possibly some of the abnormal results stated in the paper might be thus explained.

Mr. RICHMOND said that it was quite true that his opinions had changed as to the extent to which the fat could be obtained from sour milk by the maceration process. During the past few years he had learned a good deal more about the process, and had come to the conclusion that formerly he had not applied it in the best way. It was important to dry the milk to the right extent and to grind it thoroughly. They had found about a day and a half's hard grinding necessary. He quite agreed with Mr. Lloyd as to the difficulty caused by gas-producing organisms. Nevertheless, he thought that even in that case, by determining a sufficient number of compounds, the composition of the original milk might be fairly accurately deduced. From the figures they had given it would be seen that their experience was quite at variance with Mr. Lloyd's as to the total loss being equal to half the quantity of lactic acid. He thought that merely to correct for lactic acid would by no means be sufficient. With regard to the conversion of milk-sugar into lactic acid, it was of course the case that, even in samples that had undergone what they had called normal decomposition, small quantities of volatile acids and alcohol were always produced, and therefore the very simple formula referred to would not, strictly speaking, represent what took place. Nevertheless, for purposes of correction, when each of the other substances was duly taken into account, it did not involve a serious error to apply the correction in the simplest way by assuming that the residue of the lactic acid was derived from milk-sugar according to the formula.

Dr. VOELCKER suggested that, if the authors of this paper, and also Mr. Lloyd, continued their observations, it would be advantageous to pay attention more particularly, not to normal samples, which gave the public analyst no difficulty, but to samples which were mixed, or believed to be mixed, with smaller or larger quantities of water.

Mr. ESTCOURT said that, according to the experiment at the Government Laboratory, the loss of solids-not-fat which occurred on keeping a milk was mainly due to the formation of alcohol. Indeed, the alcohol formed was said to represent seven-tenths of the total loss by keeping.

The figures given by the authors of the paper do not bear out this theory. How does Mr. Richmond account for this?

Mr. RICHMOND said that the statement that the alcohol correction amounted to seven-tenths of the total correction was to be found in the Appendix to the Report of the Departmental Committee. They did not endorse it; it was not generally correct.

Mr. LLOYD remarked that he had never found anything like 2 per cent. of acid, and only once over 1½ per cent., even in samples that had been kept for months.

The PRESIDENT said that in some samples which he had analysed some years ago he had found 1½ per cent. of total acidity, including CO_2.

Mr. RICHMOND said that in one of their samples, as would be seen, the total acidity was 2¾ per cent., and in two others it was even a little more. That, of course, was the total acidity from all sources, calculated as lactic acid. The quantity of volatile acids in those three cases was considerable, and there was very little actual lactic acid. If all the acidity were due to lactic acid, he quite agreed with Mr. Lloyd that it would not be likely to reach 1½ per cent. It was only when some unusual form of decomposition had occurred, in the course of which large quantities of other acids were produced, that these high acidities were found.

NOTE BY DR. THORPE.—Mr. Droop Richmond has courteously afforded me the opportunity of perusing the foregoing paper and of appending any comments that it might seem desirable to make. Anything like detailed criticism, however, would here be out of place; and beyond congratulating the authors upon having dealt in an instructive way with an interesting matter, I think I need only remark that the principal matter on which Messrs. Richmond and Miller's procedure differs from that of the Government Laboratory is the method of dealing with the volatile acids. In actual practice, however, we find the cases extremely rare in which the volatile acids exceed 0·2 per cent., so that the extended process described in the paper would scarcely ever be applied.

On the whole, it appears that although there are certain points open to discussion, the conclusion of Messrs. Richmond and Miller is that in general a substantially accurate determination of the original solids of the milk can be made by the method in use at the Government Laboratory. That in certain cases a somewhat closer approximation can be obtained by elaborated processes is no doubt true, and the authors are to be congratulated upon their careful investigation of the question.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Sodium Sulphite in Food. C. D. Holley. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 993-997.)—The amount of sulphite recovered from meat by distillation with phosphoric acid is found to be approximately one-fourth that originally present. Of forty-three samples of pork-sausage and Hamburg steak examined, thirty contained sulphite, the quantity actually recovered varying from 0·023 to 0·361 per cent. sodium sulphite. The amount of sulphite in fried meat is almost as great as in the uncooked state. Dried prunes, peaches, and apricots yielded considerable quantities of free or combined sulphurous acid, one sample of prunes containing as much as 0·226 per cent. calculated as sodium sulphite. W. H. S.

A New Unsaturated Fatty Acid in Japanese Sardine Oil. M. Tsujimoto. (*Journ. Coll. Engineering*, University of Tokio, 1906, iv., 1-9.)—It is stated that the Japanese sardine oil of commerce is liable to contain more or less of other fish oils, but the author has analysed three specimens which were undoubtedly derived from *Clupanodon melanosticta*. They were greenish-brown to reddish-brown in colour, had a fishy odour and taste, and on standing at low temperature yielded large deposits of "stearin." Apparently they were free from any considerable amount of unsaponifiable matter. The following analytical values were obtained:

Oil from	Specific Gravity at 15·5° C.	Acid Value.	Free Acids as Oleic Acid, Per Cent.	Saponification Value.	Iodine Value (Wijs).	Refractive Index at 20° C.	Melting-point of Fatty Acids, ° C.
Chita -	0·9347	1·32	0·66	195·76	180·7	1·4808	35·4
Chosi -	0·9318	8·22	4·13	196·16	180·57	1·4802	36·2
Hakodaté -	0·9316	5·15	2·59	194·81	187·27	1·4807	35·8

The mixed fatty acids of the three oils on treatment in ethereal solution with an excess of bromine yielded 47·09, 44·24, and 44·88 per cent. respectively of white insoluble products, which turned black at 200° C. and decomposed before their melting-point was reached. They were found to contain from 69·95 to 70·16 per cent. of bromine, and the other results of an elementary analysis also showed that they were octobromides with the formula $C_{18}H_{28}Br_8O_2$, evidently derived from an unsaturated acid, $C_{18}H_{28}O_2$, belonging to the series $C_nH_{2n-8}O_2$. The author terms this new acid *clupanodonic acid*, and calculates that it forms from 13·34 to 14·20 per cent. of the mixed fatty acids from Japanese sardine oil. By reducing it with zinc dust and alcoholic hydrochloric acid he isolated about 25·7 per cent. of the theoretical quantity of the free fatty acid, which was a yellow liquid with a fishy odour, which became oxidized on exposure to the air, and formed a dry varnish in a few days. It had an iodine value of 344·42, as compared with the theoretical value 367·73. Since no hexabromide insoluble in ether was found, the conclusion is arrived at that the presence of jecoric acid in Japanese sardine oil is doubtful. C. A. M.

TOXICOLOGICAL ANALYSIS.

A New Method of Preparing Crystals in the Examination of Blood Stains. Sarda and Caffart. (*Comptes Rendus*, 1906, cxliii., 251, 252.)—A drop of a solution of blood or of the extract from the stain obtained with the usual precautions is slowly evaporated on a glass slide at a moderate heat. The residue is then treated successively with a drop of chlorine water, a drop of pyridine, and a drop of ammonium sulphide, and after a cover-glass has been placed over it the slide is examined under the microscope with a magnification of 500.

In the presence of blood an immense number of chlorohæmatin crystals will be seen; they are rhomboidal, of an intense red or reddish-brown colour, and are grouped in crosses or stars with some isolated crystals. Groups of the red crystals of hæmochromogen will also be observed. The oxyhæmoglobin of the blood is transformed under the influence of the pyridine and ammonium sulphide into alkali hæmatin, and then into reduced hæmatin or hæmochromogen, which unites with the chlorine to form chlorohæmatin. The author has obtained excellent results with stains ten years old, and recommends this method as preferable to any of the ordinary methods. A drawback is that the crystals do not keep long when exposed to the air, and should therefore be mounted in Canada balsam. C. A. M.

ORGANIC ANALYSIS.

Röse-Herzfeld and Sulphuric Acid Methods for Determination of Higher Alcohols. V. H. Veley. (*Journ. Soc. Chem. Ind.*, 1906, xxv., pp. 398-401.)—From a full examination of the two methods, working with several of the higher alcohols, the author concludes that by adopting all necessary precautions as to determination of specific gravity of the spirit, calibration of the apparatus, keeping the chloroform sheltered from the light, etc., the Röse-Herzfeld method is sufficiently accurate for all commercial and legal purposes. The removal of aldehydes from the distillate from brandy or other spirit by heating for one hour with caustic soda under a reflux condenser is considered more satisfactory than the sulphuric acid process and subsequent destruction of the aldehydes by means of *m*-phenylene-diamine, or preferably aniline phosphate. The line of demarcation between the chloroform and aqueous layer is more distinct if, after shaking, the chloroform is allowed to drop very slowly from the upper to the lower bulb, and Grünheit's observation is confirmed that after the manipulation has been completed the chloroform slowly contracts, sometimes requiring an hour before a constant level is reached.

Though less accurate, the sulphuric acid process gives valuable information. The risk of loss of higher alcohol before it is attacked by the sulphuric acid during the heating of the mixture in equal volumes of the sulphuric acid and alcohol solution may be obviated by using glass-stoppered bottles of about 50 c.c. capacity, the stoppers of which are tightly tied down during the heating operation. A bath of saturated NH_4Cl is suitable for this purpose, a constant temperature of 109° to 110° C. being thereby readily maintained. The colour produced varies considerably with the temperature, a difference of 2° or 3° causing a marked difference in the tint. It is better to cleanse the bottles prior to use with sulphuric acid only, not using nitric acid also, as the latter is a source of danger. From the author's experience it seems probable that no reaction would take place if the higher alcohols and acid were both pure and the containing vessel quite clean. W. H. S.

Unification of Reduction Methods of Sugar Analysis. L. S. Munson and P. H. Walker. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., pp. 663-686.)—The use of one standard set of solutions is proposed for all reducing sugar determinations, the precipitated cuprous oxide being weighed. The solutions recommended are one containing 34·639 grams of crystallized $CuSO_4$, which must not contain more than a trace of iron, per 500 c.c., and the other containing 173 grams of Rochelle salt and 50 grams of sodium hydroxide per 500 c.c. For a determination 25 c.c. of each solution are placed in a 400 c.c. beaker, and 50 c.c. of reducing sugar solution added, or if less sugar solution is used, the volume is made up to 100 c.c. with water. The beaker is covered with a watch-glass and heated on asbestos gauze over a Bunsen, the flame being so regulated that boiling begins in four minutes. Boiling is continued for exactly two minutes, and the cuprous oxide at once filtered, without dilution, on an asbestos felt in a Gooch crucible, using suction, the cuprous oxide being thoroughly washed with water at about 60° C., then with 10 c.c. of alcohol, and finally with 10 c.c. of ether, the crucible transferred to a water-oven at 100° C. for thirty minutes, cooled, and weighed. The asbestos is best prepared by first digesting with 1 : 3 HCl for two or three days, then washing free from acid and digesting for a similar period with soda solution, after which it is treated for a few hours with hot alkaline copper tartrate of the strength employed in the analyses. It is then washed free from alkali, digested for several hours with HNO_3, and after a final washing is ready for use. The same felts may be used repeatedly, the cuprous oxide being dissolved off each time with HNO_3. A blank experiment should be made each day to determine the correction necessary for the slight spontaneous precipitation of cuprous oxide which takes place when copper tartrate is boiled, and also for the slight loss of asbestos by solution in the alkaline liquid. The copper-reducing powers of δ-glucose, invert sugar, and two mixtures of invert sugar and sucrose have been determined, and tables are given showing the equivalents for these of all quantities of cuprous oxide between 10 and 490 mgms. The results are summarized in the following formulæ, where y = mgms. of cuprous oxide, and x = mgms. of reducing sugar: For δ-glucose, $y = 0{\cdot}5614 + 2{\cdot}3484x - 0{\cdot}001209x^2$; for invert sugar, $y = -0{\cdot}2460 + 2{\cdot}2747x - 0{\cdot}001077x^2$; for invert sugar and sucrose, 0·400 gram total, $y = 6{\cdot}3886 + 2{\cdot}2279x - 0{\cdot}0009703x^2$; and for invert sugar and sucrose, 2·000 grams total, $y = 20{\cdot}6600 + 2{\cdot}2021x - 0{\cdot}0009030x^2$.

W. H. S.

The Determination of Sugar in Urines containing little Glucose. J. Blaise. (*Ann. de Chim. Ann.*, 1906, vol. 11, pp. 285-287.)—Most urines poor in glucose give, when boiled with Fehling's solution, a yellowish-green precipitate, which remains in suspension and prevents the exact observation of the end-point of the reaction. The simplest method of obviating this is to add a known quantity of glucose in the following manner: 10 c.c. of the copper solution and 10 c.c. of the alkaline solution (corresponding to 0·05 gram of glucose) are mixed with 2 c.c. of a solution containing 0·01 gram of glucose, and boiled. The copper oxide is immediately precipitated, and the operation is then continued in the usual way with the urine to be examined.

C. A. M.

The Determination of Nitrosophenol. L. Lemaire. (*Bull. Soc. Chim. Nord France*, 1906, 64; through *Ann. de Chim. anal.*, 1906, vol. 11., pp. 262, 263.)—The method is based on the fact, observed by Spitzer, that on gently heating a solution of phenylhydrazine with a solution of a nitroso compound in glacial acetic acid nitrogen is liberated in accordance with the following equation:

$$R.NO + C_6H_5N.NH_2 = R.N + C_6H_6 + H_2O + N_2.$$

Commercial nitrosophenol is a brown substance, usually moist, and containing variable amounts of impurities derived from the reagents used in its manufacture. In making an analysis 2·5 grams of the well-mixed sample are incorporated little by little with glacial acetic acid, and the whole made up to 100 c.c. with the same acid. The apparatus used by the author is a 200 c.c. flask, closed with a stopper, in which are three openings. Through one of these passes a tube for the introduction of a current of carbon dioxide; the second receives a bromine bulb, the opening of which communicates by means of a T-tube with a carbon dioxide supply-tube, whilst the third is connected with a condenser. A tap is placed between the T-tube and the flask, so that the carbon dioxide can be passed directly into the flask or by way of the bromine bulb. The condenser is connected with a Dupré's apparatus, containing a solution of potassium hydroxide (1 : 2). Twenty c.c. of the solution of nitrosophenol (= 0·5 gram) and 20 c.c. of glacial acetic acid are placed in the flask, and the air expelled first from the flask and then from the bromine bulb by means of the carbon dioxide. The refrigerator is then connected with the collecting vessel, and the expulsion completed by means of a slow current of the gas. A solution of 2 c.c. of phenylhydrazine in 40 c.c. of acetic acid is now introduced into the bromine bulb, the flask gently heated, and the evolved nitrogen collected, measured, with the usual corrections for temperature, etc., and calculated into NO. The percentage of nitrosophenol, $NO.C_6H_4.OH$, in 0·5 gram of the substance can be obtained by means of the formula:

$$x = 1{\cdot}449 \times V^{\circ} (b - w)$$

where V° represents the volume of nitrogen at 0° C., b represents the atmospheric pressure, and w the tension of water vapour, plus the tension of benzene vapour in mgms. of mercury at the temperature t. C. A. M.

Direct Estimation of Nitroglycerine in Cordite, etc. O. Silberrad, H. A. Phillips, and H. J. Merriman. (*Journ. Soc. Chem. Ind.*, 1906, xxv., 628-630.)—The method is based on the reduction of the saponification products of nitroglycerine

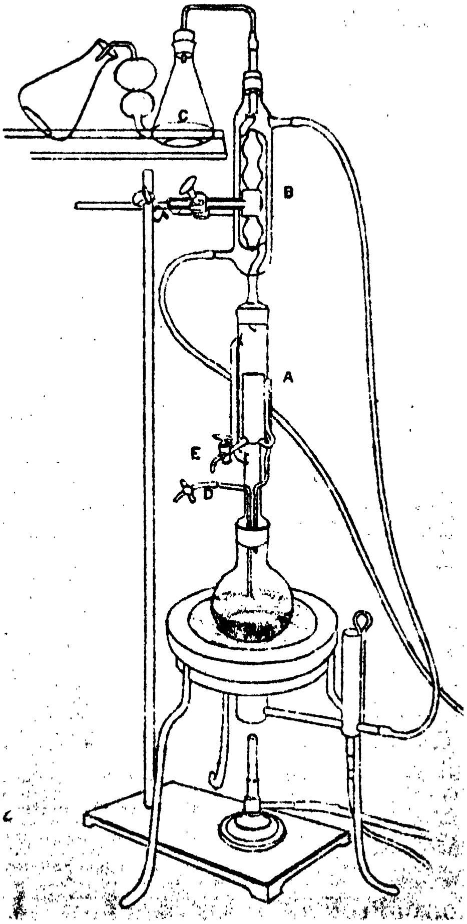


Fig. 1.

to ammonia with zinc, iron, and caustic soda. A weighed quantity of the ground cordite, sufficient to yield about 2 grams of nitroglycerine, is placed in a thimble in the Soxhlet retractor A, which is fitted up as shown in Fig. 1. Eighty c.c. of absolute ether is poured into the flask, and extraction carried out in the usual way. When extraction is complete, the thimble containing the residual nitrocellulose is washed with a little fresh ether, and removed from the extractor. The absorption flasks C, containing 10 c.c. of $\frac{N}{10}$ acid, are now affixed, and about 50 c.c. of a solution prepared by dissolving 5 grams of sodium in 100 c.c. of absolute alcohol run into the flask little by little through the side tube D. Reaction rapidly takes place, and is completed by warming on the water-bath for about six hours, after which the ether is distilled up into the Soxhlet and run off by means of the tap, the residue dissolved in water and made up to 250 c.c., the aqueous Soxhlet and ether washings being also added to the solution. Fifty c.c. of this solution are then transferred to the flask F (Fig. 2), 50 grams of a mixture of powdered zinc (2 parts) and reduced iron (1 part) added, together with 50 c.c. of 40 per cent. sodium hydroxide solution, and the ammonia distilled off in a slow current of air and collected in about 75 c.c. $\frac{N}{10}$ acid in the absorption flask H, the excess of acid being titrated back. Each c.c. of $\frac{N}{10}$ acid corresponds to 0·00757 gram of nitroglycerine. W. H. S.

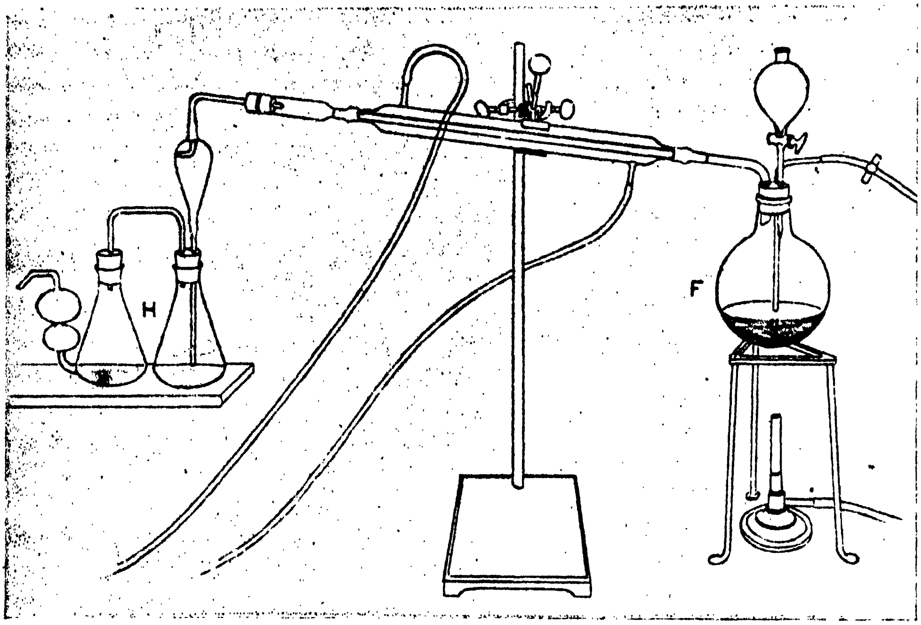
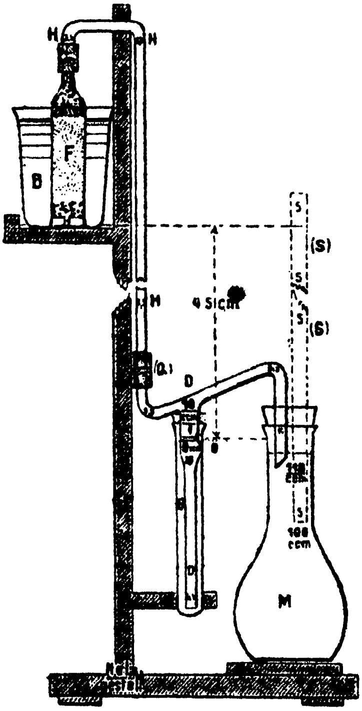


FIG. 2.

The Determination of Proteids by Means of Acetone. F. Bordas and Toutplain. (*Comptes Rendus*, 1906, cxlii., 1345, 1346.)—Proteids, such as albumin, casein, fibrin, and gelatin, are insoluble in acetone, whether pure or diluted with a suitable quantity of water. Diastases and peptones are also precipitated by acetone, which is thus a good reagent for the separation of fats from proteids. When the precipitation is made in the cold from a proteid solution, which should be neutral or nearly so, the liquid separated from the precipitate by means of centrifugal force will contain only a trace of nitrogen. In the case of cheese 2 grams are ground up with 5 to 10 c.c. of water and 30 to 35 c.c. of acetone added, little by little, with continual stirring. The insoluble proteids are collected, washed with dilute and finally with pure acetone, dried, weighed, ignited, and the amount of ash deducted. Milk is analysed by treating 10 c.c. with 20 c.c. of acetone, shaking the mixture to effect complete precipitation, separating the precipitate in a centrifugal machine and treating it as above described. For butter 10 grams are extracted with pure acetone and the residue treated with aqueous acetone, which leaves the pure casein. C. A. M.



On the Execution of the Comparative Analyses with "gewachsene" (porous) Alumina. Wislicenus. (*Ledermarkt, Collegium*, 1906, 77, 87; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 167.)—The comparative analysis with "porous alumina" (*v.* ANALYST, 1904, xxix., 377) had to be delayed until a uniform material could be made on the large scale, which has now been done by E. Merck. In the meanwhile it has been shown that the absorption of the tannin by the alumina increases with increasing dilution of the solution, reaching a maximum at a concentration of 0·1 per cent. of tannin, and decreasing again below this limit. The analysis is carried out in the apparatus shown in the figure. The Allihn tube F, shortened to 7 cms., is charged with 2 grams of freshly ignited alumina, and placed in the beaker B, which contains the tanning solution diluted to twice its original volume. The vessel G serves to receive the first 10 c.c. of the filtrate, which are not always quite clear; the tube S automatically interrupts the filtration when 110 c.c. have passed over, which should require from one to three hours; 100 c.c. of the filtrate are then evaporated and dried for the determination of the "non-tanning" materials. A. G. L.

The Detection and Determination of Syringin. J. Vintilesco. (*Journ. Pharm. Chim.*, 1906, xxiv., 145-154.)—Both the glucoside prepared by the author from the plant and the commercial product, after recrystallization from water, had the characteristics given by Koerner. They contained 4·16 per cent. of water of crystallization, melted at 190° to 192° C., and gave an intense violet-red coloration with concentrated sulphuric acid. The anhydrous syringin was found to have an optical rotation of $\alpha_D = -17°$, and on hydrolysis to yield *d*-glucose and an insoluble substance syringinin—

$$C_{17}H_{24}O_9 + H_2O = C_{11}H_{14}O_4 + C_6H_{12}O_6.$$

The author's method of determining it in extracts of the leaves, etc., of the lilac or privet is based upon its hydrolysis by means of emulsin, the insoluble syringinin subsequently carrying down the small quantities of the enzyme left in the solution. Experiments with the pure anhydrous glucoside showed that 48·387 per cent. of glucose were formed by the action of the emulsin. Hence the quantity of the glucoside in a liquid not containing any other glucoside (which was the case with lilac and privet) could be calculated by means of Bourquelot's formula,

$$q = \frac{100g}{2Rm + 105g},$$

where q represents the amount of glucose set free in 100 c.c. of liquid, and corresponding to a return of 1° to the right in the optical rotation ($L = 200$ mm.); g, the amount of glucose furnished by 1 molecule of the glucoside; R, the rotatory power of the glucoside; and m, its molecular weight ($= 372$). Thus a return of 1° to the right in a solution of syringin acted upon by emulsin corresponds to the formation of 0·570 gram of glucose in 100 c.c. of the liquid—*i.e.*, to 1·178 gram of the glucoside.

For the extraction of the syringin in quantity the author made use of the fact that hot ethyl acetate saturated with water will remove all glucosidal substances without extracting any appreciable quantity of sugars. One kilogm. of the finely-divided leaves, etc., was thrown into 3 litres of boiling water containing calcium carbonate in suspension, and the boiling continued for a few minutes to destroy any enzymes. The mass was then crushed in a machine and once more boiled in the hot water for a few moments. The liquid was then cooled, filtered, and distilled under reduced pressure until a soft paste remained. This was extracted with the hot ethyl acetate and water, and the extract on cooling left a deposit of syringin, which could be obtained pure by a single recrystallization from water.

Extracts of the fresh plant behaved differently to solutions of pure syringin on treatment with emulsin. In the latter case it was necessary to add from time to time fresh quantities of the enzyme in order to effect the complete hydrolysis of 1·22 gram of syringin in 100 c.c. of water; but this was not necessary, in most instances, with the extracts, even when they contained a much larger amount of the glucoside (*e.g.*, 3 grams in 100 c.c. in the liquid from lilac leaves, and 3·6 grams from Japanese privet). The solubility of pure syringin is about 1·4 grams in 100 c.c. of water, and as the above-named 3 and 3·6 grams were soluble in the extracts from the plants, it shows that the solubility was increased by the presence of other extractives. The emulsin can only be added to the extracts from the plants after inversion of the cane sugar by the action of invertin, and a determination of the cane sugar can thus be made simultaneously. C. A. M.

The Amount of Sulphur in Commercial Petroleum Oils. R. Kissling.

(*Chem. Rev. Fett- u. Harz-Ind.*, 1906, xiii., 157, 158.)—It was asserted by Gräfe that commercial petroleum oils were approximately free from sulphur, and in any case hardly ever contained more than 0·02 per cent. This is not confirmed, however, by the independent analyses of Engler and the author, who have found that a proportion of 0·05 per cent. of sulphur is by no means exceptional in commercial petroleum lamp oils, and that samples containing less than Gräfe's maximum of 0·02 per cent.

are of rare occurrence. The following are typical results: Kaiser oil, 0·01; astral oil, 0·02; ordinary Pennsylvanian petroleum, 0·027 to 0·029; Russian (Baku), 0·027 to 0·029; Galician, 0·039 to 0·061; American (Ohio), 0·039 to 0·05; and German (Alsace), 0·05 to 0·068 per cent. of sulphur. C. A. M.

The Examination of Turpentine Oils. Utz. (*Chem. Rev. Fett- u. Harz-Ind.*, 1906, xiii., 161-163.)—A determination of the bromine value was advocated by Vaubel (ANALYST, xxxi., 200) as a means of judging of the purity of turpentine oil. From 1 to 2 grams of the sample were dissolved in chloroform, the solution mixed with 100 c.c. of water, 5 grams of potassium bromide and 10 c.c. of hydrochloric acid, and titrated with a standard solution of potassium bromate. The absorption of bromine was regarded as complete after thirty minutes, and the absorbed bromine was calculated as having combined with the pinene to form a tetrabromide, any further decomposition of this being neglected:

$$C_{10}H_{16} + 4Br = C_{10}H_{16}Br_4,$$

according to which 100 grams of pinene absorb 254 grams of bromine. In general, commercial turpentine oils were found to have a bromine value of 220 to 230 corresponding to 86·6 to 90·5 per cent. of pinene, whilst the limits were 204 and 240. In the author's experiments with this method, the following values were obtained: Turpentine oils, 222·2 to 241·2; pine oils, 196·8 to 197·4; double rectified pine oil, 197·4; pinoline (3), 37·68 to 90·48; rosin oils, 65·22 to 78·24; dark rosin oil, 113·70. Turpentine substitutes: Ultra-turpentine oil, 11·22; terpentane, 9·6; hallol, 107·0. From these results the author concludes that the variation in genuine oils is so great that the method cannot be relied upon as a test for the purity of turpentine oil, although it may be found of value in determining whether or no a given substitute contains pinene.

Indian Turpentine Oil.—A sample obtained from *Pinus longifolia* (Roxb.) gave the following results: Specific gravity, 0·8734; optical rotation, +3° 13′; solubility in 90 per cent. alcohol, 1 in 7 or 8; and refraction at 15° C., 1·4788. C. A. M.

The Characteristics of Owala Oil. K. Wedemeyer. (*Chem. Rev. Fett- u. Harz-Ind.*, 1906, xiii., 210, 211.)—The seeds known as "Owala" are obtained from *Pentaclethra macrophylla*, a tree belonging to the *Mimoseæ*, and growing on the West Coast of Africa. The brilliant chestnut brown shells are flat and oval in shape. One kgm. of the fruit yielded, on the average, from 8 to 20 grams of seeds, 20·6 per cent. of which was shells and 79·4 per cent. kernels. The entire fruit yielded 30·4 per cent. of oil on extraction with ether and contained 29·39 per cent. of proteids, whilst from the kernel alone 41·6 per cent. of oil was extracted, and the residual pulp contained 48·25 per cent. of proteids. The extracted oil had a slight yellow colour, and was fluid at the ordinary temperature though giving a slight deposit. It had a pleasant taste with an after bitterness and an aromatic odour. When refined it gave a fine oil, which would be suitable for food purposes. The following analytical results were obtained: Specific gravity at 25° C. = 0·9119; refractometer reading (Zeiss) at 40° C. = 59·2°; Hehner value, 95·6; Reichert-Meissl value, 0·6; saponification value, 186·0; iodine value, 99·3; acid value, 9·0; acetyl value, 37·1; unsaponifiable matter, 0·54 per cent.; melting-point of fatty acids, 53·9° C.; and solidification-point of fatty acids, 52·1. The oil itself gave a white flocculent deposit when cooled to 18° to 19° C., and at 4° C. became a butter-like mass. C. A. M.

The Occurrence of Clupanodonic Acid in Herring and Whale Oils. M. Tsujimoto. (*Journ. Coll. Engineering*, University of Tokio, 1906, iv. 11-14.)—Two samples of herring oil from *Clupea pallasi* and one of whale oil from *Rhachianectes glauca* have been examined by the author. The fatty acids from the herring oils yielded 21·7 and 12·68 per cent., and those from the whale oil 27·8 per cent. of white bromides insoluble in ether, and turning black before melting. All three contained from 69·23 to 70·12 per cent. of bromine ($C_{18}H_{28}Br_8O_2$ requires 69·84 per cent. of bromine), and the amount of carbon and hydrogen also agreed with the composition of clupanodonic octobromide. Hence the author concludes that clupanodonic acid is an important constituent of these oils, though the amount (3·82 to 8·39 per cent. of the mixed fatty acids) is much less than in the case of Japanese sardine oil. No evidence could be obtained from an examination of the bromine compounds of the two highly unsaturated fatty acids, $C_{20}H_{32}O_2$ and $C_{24}H_{40}O_2$, found by Bull (*Chem. Zeit.*, 1899, 996) to be present in herring oil. C. A. M.

The Detection of Inosite in Urine and other Fluids. G. Meillière. (*Tribune Médicale;* through *Ann. de Chim. Anal.*, 1906, vol. 11, pp. 294, 295.)—The following method is recommended as giving better results than any of those ordinarily used: 25 c.c. of the urine are acidified with 2·5 c.c. of glacial acetic acid, and treated with about 2·5 c.c. of a saturated solution of barium nitrate and the same quantity of a solution of lead nitrate (1 : 5), followed by 3 to 8 c.c. of a 10 per cent. solution of silver nitrate. The precipitated silver chloride is separated with the aid of centrifugal force, and the clarified liquid treated with ammonium hydroxide until a persistent turbidity appears, then rendered distinctly alkaline with 12 drops of ammonium hydroxide, and heated gently with 3 c.c. of lead subacetate solution (strength not given). The resulting precipitate, which will contain the whole of the inosite, is separated by centrifugal force, triturated in 25 c.c. of water containing 5 drops of ammonium carbonate solution, and again separated in the centrifugal machine. It is next suspended in 25 c.c. of water, and treated with hydrogen sulphide, the liquid evaporated to about 2 c.c., and, after the addition of 20 c.c. of alcohol and 15 c.c. of ether, allowed to stand for some time, after which it is whirled. Subsequently the deposit is washed with 2 or 3 c.c. of water, which is separated in the same way, to remove the uric acid, and the inosite can thus be obtained in a crystalline state. Obviously, if the urine contained albumin, the latter must be removed before testing for inosite, whilst any sugar must be separated by means of fermentation. Inosite can be identified by treating it with nitric acid, which converts it into an acid the cadmium and strontium salts of which have characteristic colours. Another test is to evaporate the inosite with a small quantity (10 drops) of acid mercuric nitrate, a red colour being obtained as soon as the residue is dry. The reagent is prepared by mixing 10 grams of yellow mercuric oxide and 20 c.c. of nitric acid with sufficient water to make up 200 c.c. The red coloration should not disappear on the addition of glacial acetic acid, and on then adding 3 c.c. of water and 5 drops of a 20 per cent. solution of strontium acetate and warming, the liquid should change to a dichroic rose tint recalling that of eosin solutions. According to the author, inosite is a frequent constituent of urines, especially of those that give an abnormal reduction when heated with Fehling's solution, with a green precipitate and apparently no deposit of cuprous oxide. C. A. M.

Extraction of Fat from Fæces, and the Occurrence of Lecithin. J. H. Long. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., pp. 704-706.)—The fat in fæces is found to be more completely extracted by spreading 10 to 15 grams of the sample (after moistening with very dilute HCl to decompose any soaps) over a strip of paper, rolling up the paper, drying, and extracting in a Soxhlet apparatus for two days with anhydrous ether, than by drying, grinding with sand, and extracting from a paper shell in a Soxhlet for the same period. The amount of phosphates determined in the extract after evaporation of the ether amounted, in three samples, to 1·35, 0·84, and 1·26 per cent. P_2O_5 respectively on the dry fæces, which, calculated as lecithin, would correspond to a daily excretion of lecithin of 3 to 5·5 grams. No evidence could be obtained of any pathological condition in the man furnishing the material examined, and two further samples of fæces of the same individual tested some weeks later yielded 0·294 and 0·298 per cent. P_2O_5 respectively, corresponding to an excretion of about 1 gram of lecithin daily. W. H. S.

INORGANIC ANALYSIS.

On the Analysis of the Metals of the Platinum Group. N. A. Orlow. *Chem. Ztg.*, 1906, xxx., 714.)—Platinum, iridium, ruthenium, and rhodium, act catalytically on hydrogen peroxide; osmium, on the other hand, is dissolved by hydrogen peroxide solutions, as is also osmium hydroxide, $Os(OH)_4$, which is oxidized to the tetroxide. Histological preparations blackened by osmic acid are decolourized by hydrogen peroxide, the osmium going into solution. The reaction may be used to separate osmium from the other platinum metals.

Palladium chloride, $PdCl_2$, acts on silver iodide with formation of the black insoluble palladium iodide. The chlorides of the other platinum metals do not show this reaction, which may be used to separate palladium from the other metals. The palladium may be recovered from the precipitate by treatment with potassium iodide or thiocyanate, or else by igniting it and treating the residue with aqua regia.

A. G. L.

Apparatus for the Determination of Arsenic. A. Kleine. (*Chem. Ztg.*, 1906, xxx., 585.)—The apparatus consists of a flask for the determination of arsenic by distillation. The flask has an upwardly-inclined side exit-tube, bent down sharply near its end, and ground into the funnel-shaped top of a vertical spiral condenser; the joint is made absolutely tight by keeping a little water in the funnel. The flask itself has a funnel-shaped top, with a central tube reaching to the bottom of the flask, which is closed during the distillation by means of a ground-in glass rod, this joint also being made quite tight by keeping some liquid in the funnel. The apparatus is made by Ströhlein and Co., of Düsseldorf. A. G. L.

A New Method of Separating Antimony and Tin. A. Czerwek. (*Zeit. anal. Chem.*, 1906, xlv., 505-512.)—The method is based upon the formation of a double compound of stannic acid and phosphoric acid which is insoluble in water and nitric acid, but is partially soluble in dilute hydrochloric acid. In order, therefore, to separate tin from antimony by converting the former into this compound it is essential that the nitric acid solution shall be completely free from chlorine ions. In analysing an alloy, 0·5 gram of the sample is treated at 40° to 50° C. with a solution consisting of 15 c.c. of nitric acid (specific gravity 1·42), 15 c.c. of water, and about 6 grams of tartaric acid, and allowed to stand in a warm place, or at the ordinary temperature, for two or three hours, when the metals will have completely dissolved. The solution is now rapidly heated to incipient boiling and treated with 5 to 30 drops (according to the amount of tin) of a 45 per cent. solution of phosphoric acid (specific gravity 1·3), being meanwhile continually shaken. It is next diluted with about 300 c.c. of boiling water and the precipitate allowed to subside on the water-bath, which will take about fifteen minutes. The clear supernatant liquid is decanted through a filter, the precipitate washed with hot water containing ammonium nitrate, and while still moist dissolved in warm ammonium sulphide; the tin is then reprecipitated from the *cold* and diluted solution by the addition of sulphuric acid. The beaker is left on the water-bath for some time, and the greenish-grey precipitate separated and washed as before. Finally it is dried, oxidized in a weighed crucible with nitric acid (specific gravity 1·42), dried on the water-bath, ignited, and weighed as tin dioxide.

The filtrate from the tin is neutralized with ammonia heated with a sufficient quantity of ammonium sulphide, and acidified with acetic acid. The precipitate of antimony sulphide is left to settle on the water-bath and filtered off while still hot. It is then washed with water containing ammonium nitrate, and dissolved in ammonium sulphide, the solution evaporated to dryness on the water-bath, and the residue oxidized in the usual way with fuming nitric acid, and weighed as antimony tetroxide.

The accuracy of the method is shown by test analyses of alloys containing known quantities of antimony and tin. In the case of alloys containing other metals in addition the tin is precipitated as described above, and the filtrate neutralized and treated with sodium or ammonium sulphide solution, which precipitates the other metals, whilst the antimony is separated from the new filtrate by means of acetic acid. Electrolytic precipitation from an ammonium sulphide solution has been found a suitable method of throwing down antimony and tin prior to their separation from each other. C. A. M.

A Modification of Schlagdenhaufen's Reaction for Magnesium. J. Bellier. (*Ann. de Chim. Anal.*, 1906, vol. 11, pp. 283-285.)—The following method of applying the colour reaction of magnesium salts with alkali hypoiodites is capable of detecting 1 part in 20,000: About 10 c.c. of the solution of the magnesium salt are mixed with 1 c.c. of a 1 per cent. solution of potassium iodide saturated with iodine, and the mixture shaken with 15 drops of $\frac{N}{10}$ sodium hydroxide solution. In the presence of 0·1 gram of magnesium the liquid becomes reddish-brown, and then gives a flocculent deposit of the same colour. With 0·05 gram there is only a brownish to yellowish-red coloration. Ammoniacal salts prevent the reaction altogether, as do also acids and alkalies, whilst calcium salts in large quantity render it less sensitive. The reaction cannot be used for the gravimetric determination of magnesium, owing to the solubility of the precipitate in water.

C. A. M.

Iodometric Determination of Vanadic Acid. P. Hett and A. Gilbert. (*Zeit. öffentl. Chem.*, 1906, xii., 265, 266.)—Although other chemists have found that the reaction between vanadic acid and potassium iodide in acid solution is untrustworthy, the authors state that they have obtained good results by the method. The vanadate salt or ore is fused with sodium hydroxide, the melt dissolved in water, filtered, and the filtrate diluted to a known volume. A portion of the solution is then acidified with either hydrochloric or sulphuric acid, potassium iodide is added, and the liberated iodine titrated as usual. The reaction proceeds according to the equation, $V_2O_5 + 2HI = V_2O_4 + H_2O + 2I$.

W. P. S.

A Qualitative Test for Phosphorus. Mauricheau-Beaupré. (*Comptes Rendus*, 1906, cxlii., 1206, 1207.)—This is based on the fact that glass heated to redness and brought in contact with the vapours of phosphoric acid is rendered opaque. Hydrogen or acetylene burning in a Bunsen burner gives the best flame for the purpose, but either gas must be freed from any hydrogen phosphide by passing it through sulphuric and chromic acids. The test is so sensitive that acetylene containing only 1 part in 10,000 of hydrogen phosphide makes the glass opaque. In applying the test, a piece of glass tubing, 5 to 10 mm. in diameter, is supported on a platinum wire at the top of the oxidation zone of the flame.

In the case of metals, such as iron, in which the phosphorus is liberated by the action of an acid, the impure hydrogen is conducted into the air-holes of the Bunsen burner, or it can be burnt directly from a metallic jet. Organic compounds are burnt in the upper part of the blue zone of the flame, the glass tube being heated to redness above. The reaction does not take more than one to two minutes, whilst even ten minutes' heating is insufficient to devitrify the glass in the absence of phosphorus. Glass heated in a pure flame loses in weight, whilst if phosphorus was present it gains in weight (*e.g.*, 0·001 gram on 6·94 grams). In one experiment the part of the tube rendered opaque was found to contain 0·718 per cent. of phosphorus, none being present in the unattacked glass. Hydrofluoric acid vapour when present in the gas in only small quantities does not attack glass at red heat, whilst compounds of arsenic, or antimony, or boric acid, do not give any deposits that could be confused with the phosphorus deposit.

C. A. M.

Determination of the Sodium Phosphates. C. C. Ahlum. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 533-537.)—Disodium hydrogen phosphate and trisodium phosphate are both alkaline to methyl orange, and may be titrated with hydrochloric acid, the reactions being:

$$HCl + Na_2HPO_4 = NaH_2PO_4 + NaCl;$$
$$2HCl + Na_3PO_4 = NaH_2PO_4 + 2NaCl.$$

Two methods are given for the separate estimation of the two salts in a mixture containing both, the first based on the conversion of Na_3PO_4 into Na_2HPO_4 with formation of Na_2CO_3, when CO_2 is passed through its aqueous solution, according to the equation

$$CO_2 + 2Na_3PO_4 + H_2O = 2Na_2HPO_4 + Na_2CO_3;$$

and the second depending on the fact that, when a solution containing Na_2HPO_4 and Na_3PO_4 is acidified, and then neutralized with Na_2CO_3, adding an excess of the latter, the resulting solution contains Na_2HPO_4 and Na_2CO_3. In both cases the total alkalinity of the original mixture is titrated with $\frac{N}{10}$ HCl before treatment.

In the first method, 2 grams of the mixed salts are dissolved in water, and CO_2 passed through the solution until reaction is complete (about ten to fifteen minutes). The solution is then evaporated to dryness, and the Na_2CO_3 in the residue estimated in the Schrötter apparatus, the amount of CO_2 evolved multiplied by 7·4545 giving the amount of trisodium phosphate present. The number of c.c. $\frac{N}{10}$ acid, equivalent to the trisodium phosphate, is readily calculated from the above equation, and this, deducted from the number of c.c. used in the titration of the original, gives the number required by the disodium hydrogen phosphate, and this, multiplied by 0·0142, gives its amount.

For the alternative method, 1 gram is dissolved in 50 c.c. water containing a drop of methyl orange. A slight excess of HCl is added, and the solution boiled for ten minutes, after which an excess of Na_2CO_3 is added, and the solution concentrated as far as possible by boiling, transferred to a tared platinum dish, evaporated on a steam-bath, dried, and weighed. The mass is then pulverized with a porcelain pestle, halved, and the CO_2 determined in one half by the Schrötter apparatus, the other half being titrated with $\frac{N}{10}$ HCl. The difference between the number of c.c. acid used in the titration of the original mixture and that used in the latter titration, minus the number of c.c. equivalent to the Na_2CO_3 found by the Schrötter apparatus, represents the loss in alkalinity due to the conversion of the Na_3PO_4 into Na_2HPO_4, and this, multiplied by 2 × 0·0082, gives the amount of Na_3PO_4, the difference between the number of c.c. of acid equivalent to the Na_3PO_4 and that required by the original, multiplied by 0·0142, giving the amount of Na_2HPO_4.

W. H. S.

Methods for the Determination of Carbonic Acid. W. Holtschmidt. (*Chem. Ztg.*, 1906, xxx., 621.)—The author uses acid potassium tartrate to decompose the carbonate, the evolved carbon dioxide being either weighed after absorption by soda-lime, or titrated after absorption by caustic alkali. The acid potassium tartrate acts on carbonates only at the boiling temperature, and hence vigorous and continued boiling is necessary to insure complete decomposition. In order to obviate the difficulties caused by the large amount of condensed water obtained, the author has devised special and complicated forms of apparatus, by the use of which very good results are obtained. A. G. L.

Difficulties in the Determination of Carbon Monoxide in Gaseous Mixtures. A. Gautier and Clausmann. (*Bull. Soc. Chim.*, 1906, xxxv., 513-519.)—In analysing a mixture of nitrogen or air with carbon monoxide, or of nitrogen and various combustible gases with carbon monoxide, it is not possible to recover the whole of the latter gas, either by explosion in the presence of oxygen, or by washing with cuprous chloride solution. Thus after two successive washings as much as 1 per cent. of carbon monoxide may be left in the residual gas, whilst from 0·3 to 0·5 per cent. may remain after explosion of the mixed gases in the eudiometer. If, however, the gases left after the explosion or after the washing (diluted with air or not according to circumstances) be made to circulate through a tube containing iodic anhydride heated to 70° C., the last traces of carbon monoxide are oxidized and an exact determination can be made. In the ordinary method the small amount of carbon dioxide which may be found in the eudiometer after explosion of the gases left from the washings with potassium hydroxide, bromine, and cuprous chloride, might lead one to infer the presence of saturated gases, such as methane, in the original mixture, whereas it might really be due to the oxidation of the carbon monoxide that had not been removed by the cuprous chloride. The apparent volume of the residual nitrogen is increased by the uncombined carbon monoxide and that formed from the pyrogallol, but is reduced by the slight oxidation of the nitrogen itself during the explosion in the eudiometer. C. A. M.

Determination of Hydrogen Peroxide. W. E. Matthewson and J. W. Colvin. (*Amer. Chem. Journ.*, 1906, vol. 36, pp. 117-123.)—Hydrogen peroxide may be estimated by diluting with water until the solution is approximately $\frac{N}{5}$, and adding this solution from a burette to about 2 grams of ferrous ammonium sulphate dissolved in a little water, together with a few grams of ammonium sulphate and some phosphoric acid, a solution of titanium, which gives a deep yellow colour with the slightest excess of H_2O_2, being used as indicator. The titanium solution is prepared by fusing the dioxide with about ten times its weight of $KHSO_4$, dissolving the fused mass in cold dilute H_2SO_4, and filtering. An attempt to determine sodium nitrite by titrating with a standard solution of H_2O_2 gave slightly too high results, and for accurate results the titre of the peroxide solution should be fixed with a known amount of nitrite. W. H. S.

The Use of Sodium Hydrosulphite in Gas Analysis. H. Franzen. (*Berichte*, 1906, xxxix., 2069-2071.)—A solution of sodium hydrosulphite is recommended as an absorbent for oxygen in gas analysis. The absorption of the gas takes place in accordance with the equation—

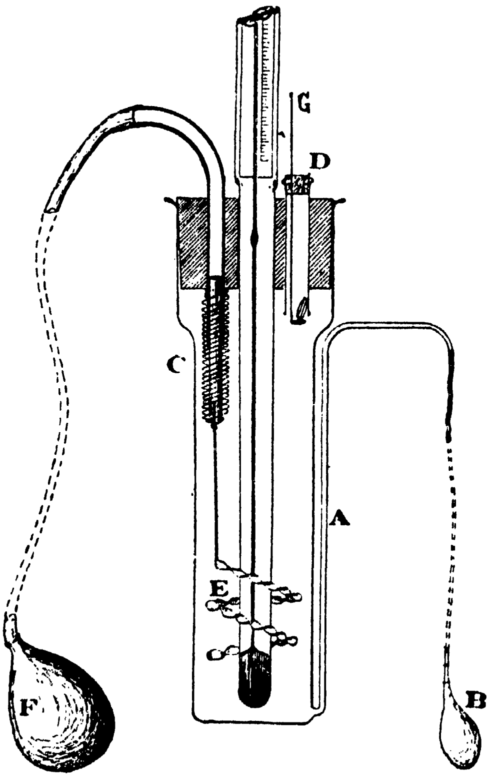
$$Na_2S_2O_4 + H_2O + O = 2NaHSO_3,$$

according to which 64 c.c. of oxygen enter into combination with 1 gram of the salt. A suitable solution for gas analysis is obtained by mixing a solution of sodium hydrosulphite (50 grams in 250 c.c.) with 40 c.c. of a solution of 500 grams of sodium hydroxide in 700 c.c. of water. One c.c. of this solution will absorb 10·7 c.c. of oxygen, the absorption being as rapid at low as at higher temperatures. The reagent, which is cheaper than pyrogallol, can be used in the analysis of mixtures of gases containing carbon monoxide and dioxide. C. A. M.

APPARATUS.

Improved Beckmann Apparatus for Molecular Weight Determinations.

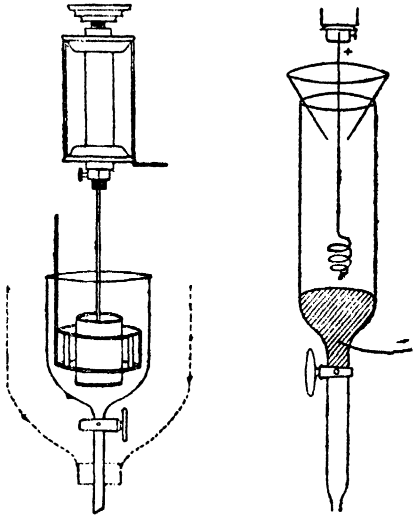
J. McC. Sanders. (*Proc. Chem. Soc.*, 1906, xxii., 165.)—The difficulty of equalizing the overcooling for parallel observations with the solvent and solution, and in the addition of ice crystals for inducing congelation, in the ordinary Beckmann apparatus is overcome by the use of a modified form of inner tube.



A narrow, almost capillary tube (A) is fused into the bottom of the solution tube, and attached to a thin rubber tube and small rubber bulb (B). The contained tube is placed in an outer jacket the wall of which is in contact with the side tube A. The liquid in A is first completely expelled by compressing B, and the rubber tube pinched while B is removed, allowed to fill itself with air, and replaced, when on releasing the rubber tube a small amount of solvent rises in A, which, in operation, being nearer to the source of cold, freezes first, and at the moment desired a mass of crystals can be projected into the main body of the solvent by a sharp compression of the bulb B. C is a pneumatic stirrer, an adaptation of the photographic "shutter release," the piston and cylinder being of aluminium or glass, ground to fit perfectly, and the agitator E being made by flattening and twisting a piece of platinum wire, which is, then coiled into a spiral form, and the other end of the wire attached to the piston. The spring is of hard-drawn aluminium or platino-iridium wire. A small vertica tube (D) passing through a third hole in the rubber stopper replaces the ordinary side tube of the Beckmann apparatus, and serves for the introduction of the substance. The apparatus can be adapted from a small Soxhlet extraction tube by cutting off the upper and lower parts and the wide side tube, and bending the narrow siphon into the form of A. W. H. S.

A Simple Form of Rotating Electrode for Electro-chemical Analysis. F. Mollwo Perkin.

(*Chem. News*, 1906, vol. 93, p. 283.)—In the apparatus shown in the figure, the vessel in which the electrolysis is carried out consists of a tap-funnel, which allows of easy washing of the cathode without interrupting the current. The electrodes are made of platinum alloyed with 20 per cent. of iridium, which forms a very rigid alloy. They are of wire 1 mm. in diameter; the anode consists of a few wide turns, whilst the cathode has a large number of turns, and a total surface of 23 square cms. The electrodes may preferably be sand-blasted. A nickel wire may also be used as cathode and will last for a large number of determinations, being but little attacked by the nitric acid used in removing the deposited zinc, copper, etc. In six copper analyses made with this apparatus the error ranged from 0 to 1·3 mgms., on from 0·05 to 0·13 gram of copper.



The author mentions that Johnson, Matthey and Co. now supply iridium of 99 per cent. purity; the metal is as hard as steel, unattacked by boiling aqua regia and by molten lead. It cannot be drawn into wire, but may be rolled into sheets whilst hot. A. G. L.

A New Burner for Spectroscopic Work. E. H. Riesenfeld and H. E. Wohlers.

(*Chem. Ztg.*, 1906, xxx., 704.)—The principle of the burner, shown in Fig. 1, consists in electrolysing the solution to be examined in the interior of a Teclu burner, the fine spray produced by the gas bubbles at the electrodes being carried up into the flame by the current of air sucked in by the gas. The burner is made in two parts, an upper one B fitting on a support C; the support also carries the vessel A, which holds 2 c.c. of the liquid to be tested, and into which dip the platinum-iridium wire electrodes. The current should have a strength of 0·2 to 1 ampère at 8 to 16 volts. For metals such as copper or thallium, which are easily deposited, a current reverser should be inserted in the circuit, and the current reversed every minute. Fig. 2 shows a convenient arrangement of the whole, B being the current reverser; the lamp used as resistance

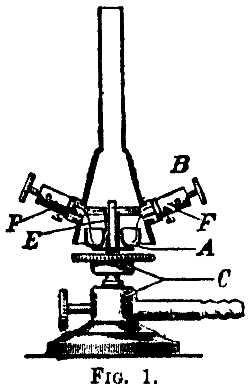


Fig. 1.

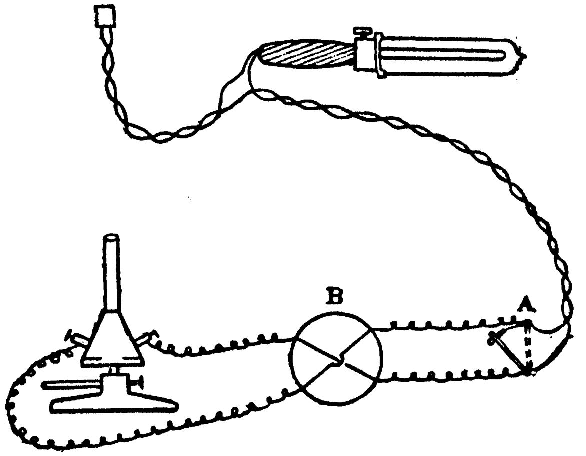


FIG. 2.

is conveniently utilized to illuminate the scale of the spectroscope. The 2 c.c. of liquid used suffice to give an intense and steady spectrum for two hours, at the end of which time the vessel will require filling up with water. The spectrum disappears as soon as the current is interrupted, the current of gas cleaning the burner at once. The burner may also be used as a convenient source of monochromatic light. The apparatus may be obtained from Franz Hugershoff, of Leipsic.

A. G. L.

* * * * *

THE SALE OF FOOD AND DRUGS ACTS: SOME DEFECTS AND SUGGESTED REMEDIES.

AN intelligent and moderately written article on the above Acts has recently appeared in the pages of our contemporary, the *Pharmaceutical Journal* of August 25, 1906, from the pen of Richard A. Robinson, junior, barrister-at-law, and assistant editor of that paper. Most of the points contended for have long been recognised by all thoughtful public analysts; indeed, so far is this the case, it is owing to this recognition that the joint committee of this Society and the Pharmaceutical was appointed to cope with cases of difficulty in drug adulterations. At the same time it should be remembered that the public analyst is not always to blame for ill-advised prosecutions; he is not the prosecutor, but is bound to analyse and report upon any sample sent him by his Authority, and, though he may give his opinion that a prosecution in a certain case would be unadvisable, this opinion may or may not be acted upon by the body under which he serves.

W. J. S.

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REVIEW.

THE ANALYSIS AND VALUATION OF OXIDE OF IRON AND LIME FOR THE PURPOSES OF GAS PURIFICATION. By H. LEICESTER GREVILLE, F.I.C., etc. (Published by Walter King. Price 2s. 6d.)

This pamphlet contains a record of the processes used for the valuation of some of the raw materials used in a gas-works, and the spent products and residuals derived therefrom. As chemist to the Commercial Gas Company, the author had exceptional opportunities of putting to the test the processes of examination which have been suggested from time to time, and in this pamphlet describes those that he adopted, as well as those of his own suggestion.

The pamphlet contains very few mistakes, and indicates that a more comprehensive work, free from debatable opinions, from the pen of this author, would be of considerable service to his colleagues.

W. J. D.

NOVEMBER, 1906. Vol. XXXI., No. 368.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An extraordinary general meeting of the Society was held on Wednesday evening, October 17, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The meeting was summoned on the instructions of the Council, for the purpose of ascertaining the views of the members in regard to the proposed incorporation of the Society under the Regulations of the Board of Trade. The Society's solicitor, Mr. Aubrey W. Rake, was present.

The notice convening the meeting having been read,

The President proposed the following resolution:

> "That in the opinion of this meeting it is desirable that the Society shall be incorporated under the Board of Trade Regulations as a limited liability company, with leave to omit the word 'Limited.'"

Dr. Thorne seconded the resolution, which was carried unanimously.

The President proposed:

> "That the draft memorandum and articles of association already submitted to the members be, and hereby are, approved by this meeting."

Mr. Kitto seconded this resolution, which was under discussion when the meeting was adjourned.

* * * * *

THE ESTIMATION OF COCOANUT OIL IN BUTTER-FAT.

By F. W. HARRIS, F.I.C.

During the past twelve months a considerable amount of experimental work has been done in my laboratory, with the object of testing the utility and reliability of the different methods for the detection and estimation of cocoanut oil in butter-fat.

The question has of late years become one of importance from a Public Analyst's point of view, especially as cocoanut oil is now placed upon the market devoid of odour and practically neutral.

At the outset experiments were made with the Bömer phytosterin acetate test.[*]

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[*] *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1904, vii., 577.

The cholesterin from butter was purified by recrystallization and examined under the microscope, after which it was converted into the acetate, and after five recrystallizations the melting-point determined.

The highest melting-point of the cholesterin acetate obtained from eight different samples was 115·4° C. (corrected).

As is well known, animal fats contain cholesterin, vegetable oils phytosterin; therefore the Bömer process, whilst affording unmistakable evidence of the admixture of vegetable oils with animal fats, cannot be employed as a means of differentiating between the adulteration of butter with cocoanut oil on the one hand and margarin on the other, as margarine almost invariably contains a vegetable oil as one of its constituents.

If by some other method the addition of cocoanut oil to butter is indicated, then the Bömer process, when used as a confirmatory test, is of great value, and if the proportion of cocoanut oil is relatively small, the confirmation by the Bömer process becomes essential.

The time occupied in carrying out this process, together with the large quantities of ether necessary, as well as the fact that at least 50 grams of the butter must be taken, precludes the possibility of this process being utilized in any other way than as a confirmatory test.

JUCKENACK AND PASTERNACK'S METHOD.*

Juckenack and Pasternack have published a method, or, more correctly, a line of reasoning, by which, from a consideration of the relationship between the Koettstorfer and Reichert-Meissl values, the presence of cocoanut oil in a sample of butter may be determined.

It is well known that the Koettstorfer and Reichert-Meissl values of butter vary in almost direct proportion to one another. In other words, the variation of these values in genuine butter is dependent upon the fluctuations in the proportion of volatile fatty acids.

Juckenack and Pasternack on this basis propose to subtract from the Koettstorfer value the arbitrary number 200, and the product thus obtained is then deducted from the Reichert-Meissl number. The formula is, therefore:

Reichert-Meissl – (Koettstorfer value – 200).

The figure thus arrived at they designate as "difference," and accordingly the value is a positive or negative + or – "difference."

For genuine butters this "difference value" must be near ± 0. In a margarine prepared without the addition of cocoanut oil the proportion of volatile acids present is relatively very small, whilst the Koettstorfer value differs but slightly from 195, so that the addition of margarine to butter would not seriously alter Juckenack and Pasternack's value.

On the other hand, cocoanut oil has a Reichert-Meissl value of about 7, and a Koettstorfer value of about 255; therefore the "difference value" of cocoanut oil becomes – 47 compared with 0 for butter.

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* *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1904, vii., 193.

The margin between these figures being considerable, it appeared to me likely to afford a ready means for indicating the presence of cocoanut oil in butter.

Obviously, the first essential in deciding the reliability of this process is to determine the variations in the "difference value" of genuine butters. With this object, I examined seventy samples of butter. The samples represented Dutch, colonial, and home produce, and the results obtained are summarized as follows:

Number of Samples.	"Difference Value."
4	−4·6 and −4·9
5	Between −4 and −3
7	Between −3 and −2
5	−2·0 and −1
10	−1 and −0
20	0 and +1
9	+1 and +2
4	+2 and +3
2	+3 and +4
2	+4 and +5
2	+5 and +6

The genuineness of these samples was determined by the Reichert-Meissl number, Koettstorfer value, refractive index, and Polenske number, and in two or three doubtful cases was confirmed by the Bömer process.

Test samples of butter were prepared, containing 5, 10, and 15 per cent. of cocoanut oil, and the analysis of these yielded the following results:

Samples.	"Difference Value."
Original butter	+ 0·5
Original cocoanut oil	−53·0
Test sample with 5 per cent. cocoanut oil	− 1·92
,, ,, 10 ,, ,,	− 4·9
,, ,, 15 ,, ,,	− 7·2

The fluctuations in the "difference value" of the above seventy butters is therefore from −4·9 to +6. Juckenack and Pasternack found the fluctuations to be between −3·5 and +4·25 for genuine butters.

It will be observed that out of the seventy samples five gave a "difference value" of from −3 to −4, whilst in the case of four samples the value lay between −4·6 and −4·9, figures identical with the test sample containing 10 per cent. cocoanut oil. In sixteen—practically 23 per cent. of the samples—the negative "difference value" was greater than −2; that is, greater than the negative "difference value" of the test sample containing 5 per cent. of cocoanut oil. It is therefore obvious that this method is useless to indicate with any certainty the presence of less than 15 per cent. of cocoanut oil.

METHODS OF POLENSKE, KIRSCHNER, ETC.

During the last two years several methods have been proposed for the estimation of cocoanut oil in butter, based on the following principles:

Firstly, that whereas the predominant constituent of the volatile acids of butter is butyric—caproic, caprylic, and capric acids being present in small quantities—the volatile acids of cocoanut oil consist principally of caprylic acid, the quantity of caproic acid being very small, whilst butyric acid is entirely absent.*

Secondly, that whereas by far the greater proportion of the volatile acids of butter-fat are soluble in water, the reverse holds good for the volatile acid of cocoanut oil.

Two methods have been proposed by Jensen and Kirschner,† in which the insolubility of the silver salt of caprylic acid is made use of. Kirschner's method, which is really a modification of Jensen's method, consists of obtaining a distillate of the volatile acids as in the determination of the Reichert-Meissl value.

One hundred c.c. of the filtered distillate are titrated with $\frac{N}{10}$ baryta, using phenolphthalein as indicator. To the thus neutralized solution, 0·5 gram of silver sulphate is added, the flask stoppered, well shaken, and allowed to stand one hour. The solution is then filtered, and to 100 c.c. of the filtrate are added 35 c.c. of water, 10 c.c. of dilute sulphuric acid, and distilled; 110 c.c. are distilled off: this should occupy one hour. The distillate is filtered, and the 100 c.c. of the filtrate titrated as before. This figure, after calculating to 5 grams of the sample, Kirschner designates as the "Neue Zahl" (new "Butter Number").

Several estimations were made with this method, but the results were unsatisfactory, probably partly from the fact that, whereas the first distillation is carried out in thirty minutes, the second distillation, according to Kirschner, should take one hour.

One point suggested by Kirschner can be utilized in conjunction with the ordinary Reichert-Meissl Number process as a qualitative test for cocoanut oil. He suggests that the remaining 10 c.c. of the original distillate be filtered, and a few drops of a concentrated silver salt solution be added. In the presence of cocoanut oil a milky solution results. Pure butter yields a clear solution, whilst margarine gives a slightly opalescent solution.

A. Muntz and H. Coudon (the ANALYST, 1905, 155) and Polenske ‡ have suggested methods based upon the difference between the ratio of soluble and insoluble volatile acids in butter and cocoanut oil.

POLENSKE'S METHOD.§

I have subjected Polenske's method to a careful and extensive examination. The process, together with the details of the apparatus, etc., have been published in the ANALYST, and are incorporated in the new edition of Lewkowitsch's "Fats and Oils," vol. ii. It will therefore be unnecessary for me to enter into any details of the *modus operandi.*

RATIO BETWEEN THE REICHERT-MEISSL NUMBER AND THE POLENSKE NUMBER.

Polenske, in his original paper, gives the results obtained by the analyses of thirty-one samples of butter. The Reichert-Meissl number of these thirty-one samples ranged from 23·3 c.c. to 30·1 c.c., whilst the Polenske number correspondingly fluctuated between 1·5 c.c. and 3 c.c. From the results obtained, Polenske concludes that the Polenske number of genuine butters—that is, the proportion of insoluble volatile acids—increases proportionately with the Reichert-Meissl number. The results tabulated by Polenske do not, however, justify this conclusion, as the following figures, taken from his original paper, indicate :

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* *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1905, ix., 65.

† *Ibid.*

‡ *Ibid.*, 1904, vii., 273.

§ *Ibid.*

Table I.

(Polenske number was given by butters with the Reichert-Meissl number.)

Polenske Number.	Reichert-Meissl Number.					
1·5	22·5	23·4				
1·6	23·3	24·5	24·8	25·1	25·2	
1·7	23·6	24·7	24·8	25·6	25·4	
1·8	25·0	25·3	26·6	25·4	26·2	26·5
1·9	25·4	26·2	26·5	26·9	27·5	
2·2	27·8	28·8				
2·5	28·8	29·5				

I found, by the examination of seventy samples of butter, that the variations in the ratio between the Reichert-Meissl number and the Polenske number was greater than is indicated in the above table.

My results may be summarized as follows :

Table II.

Reichert-Meissl Number.	Polenske Number.
23 to 24 c.c.	1·3 to 1·5 c.c.
24 ,, 25 ,,	1·4 ,, 2·2 ,,
25 ,, 26	1·6 ,, 2·3
26 ,, 27	1·9 ,, 2·6
27 ,, 28	2·5 ,, 2·9
28 ,, 29	2·4 ,, 2·9
29 ,, 30	, 3·0
30 ,, 31	2·4 , 3·3
31 ,, 32	2·4 , 3·3
32 ,, 32·5	2·6 , 3·5

The figures in this table indicate that, in a broad sense, a certain relationship exists between the Reichert-Meissl and the Polenske numbers, but that the Polenske number of genuine butters increases in direct proportion to the increase in the Reichert-Meissl number, as concluded by Polenske, is certainly not borne out.

No doubt these discrepancies in the ratio between the Reichert-Meissl number and the Polenske number are attributable to the various influences, both natural and artificial, which affect the total quantity, and probably also the ratio of the constituents of the volatile fatty acids of butter.

Accuracy of the Process.

The essential point, as I found out by experience, to obtain concordant and reliable results by this method is that the size and quantity of the pumice-stone added to promote regular ebullition should be strictly in accordance with the recommendation of Polenske.

The caprylic and capric acids, being insoluble in water, necessarily float on the surface of the liquid in the distillation flask, and, consequently, the proportion of those acids which distil over is dependent, other conditions being equal, on the state of ebullition, whether regular or spasmodic.

Regular ebullition insures that the fatty layer is thoroughly disintegrated and brought into intimate contact with the water vapour as it rises through the liquid.

In my first estimations I employed three or four pieces of pumice-stone of about 1·5 cm. diameter. The results obtained were much below those obtained by Polenske. After careful revision of the measurement of the apparatus, rate of distillation, and generally the *modus operandi*, the same low figures resulted. On again reading Polenske's original paper, I found that he recommended the use of powdered pumice-stone in quantity sufficient to cover the point of a pocket-knife blade. On repeating the estimations with this quantity of powdered pumice-stone, the results were satisfactory, duplicate estimations agreeing very closely.

In view of these results, I made a series of experiments to ascertain the extent of the effect different sizes and quantities of pumice-stone would have on the results. I found that the quantity of powdered pumice-stone recommended by Polenske was approximately equal to 0·1 gram; therefore this quantity was rigidly adhered to. The results I obtained with different sizes of pumice-stone are as follows:

Table III.

Three Pieces of Pumice-stone about 1·5 Cm.		0·1 Gram of Powdered Pumice-stone.	
Reichert-Meissl Number.	Polenske Number.	Reichert-Meissl Number.	Polenske Number.
24·8	1·4	24·8	2·2
26·8	1·5	27·4	2·5
28·2	1·5	28·6	2·4
28·2	1·8	28·8	2·5
28·3	2·1	28·6	2·9
28·6	1·8	28·7	2·9
28·7	1·8	29·2	2·9
30·8	2·1	31·3	2·55

By the use of 0·1 gram of pumice-stone the ebullition was very regular and the fatty layer thoroughly disintegrated.

After making a considerable number of estimations in duplicate, I was convinced that, provided the conditions as specified by Polenske are rigidly adhered to, the results obtained are thoroughly reliable and concordant.

As an example, I tabulate the results obtained by duplicate estimations of two samples of butter which were reported upon as containing cocoanut oil:

Table IV.

		Reichert-Meissl Number.	Polenske Number.
Sample No. 1	First estimation ...	32·2	4·0
	Second estimation ...	32·1	4·0
Sample No. 2	First estimation ...	31·0	4·1
	Second estimation ...	31·0	4·15

APPLICATION OF THE METHOD FOR THE DETECTION OF ADULTERATION OF BUTTER WITH COCOANUT OIL.

In order to ascertain to what extent this method can be depended upon to detect cocoanut oil in butters, mixtures were made containing 5, 10, and 15 per cent. of cocoanut oil. The results are tabulated in the following table:

Table V.

	Reichert-Meissl Number.	Polenske Number.	Koettstorfer Value.	"Difference Value."
Original butter	32·5 ...	2·55 ...	232·0 ...	+ 0·50
Test sample, 5 per cent. cocoanut oil	31·0 ...	3·50 ...	232·9 ...	− 1·92
Test sample, 10 " " "	30·8 ...	4·00 ...	235·7 ...	− 4·90
Test sample, 15 " " "	29·9 ...	4·80 ...	237·1 ...	− 7·20
Cocoanut oil	7·5 ...	17·60 ...	260·5 ...	− 53·00

It was my intention to extend this series of experiments very considerably, but pressure of work has prevented me.

Polenske in his paper stated that his method failed to detect less than 10 per cent. of cocoanut oil. From his exhaustive results, he concluded that generally an addition of 1 per cent. cocoanut oil increased the Polenske number by 0·1 c.c. He recommends, possibly in order to cover all natural fluctuations in the Polenske number, that before deciding whether a sample contains cocoanut oil or not the Polenske number of the sample be compared with the Polenske number of a pure butter with an equal Reichert-Meissl number. To this latter figure Polenske adds 0·5 c.c., and if the Polenske number of the sample under consideration is above this increased number, then the presence of cocoanut oil must be assumed; and in calculating the quantity of cocoanut oil present, he subtracts from the ascertained Polenske number of the sample the corresponding Polenske number for a pure butter with an equal Reichert-Meissl number, and by multiplying the product by 10 the percentage of cocoanut oil is arrived at.

In expressing an opinion as to the value of this method as a means of estimating cocoanut oil in butter without asserting that the addition of 10 per cent. of cocoanut oil could be estimated by this means, I would affirm that, provided a large number of samples of varying Reichert-Meissl numbers have been tested by this method, so as to form a reliable basis of comparison, then it is, in my opinion, quite possible to detect the admixture of 10 per cent. of cocoanut oil with a relative degree of certainty; and an addition of 15 per cent. can not only be detected, but fairly accurately estimated, by the Polenske method. The fact must not be lost sight of that, by the addition of cocoanut oil to butter, not only is the Polenske number increased, but the Reichert-Meissl number is lowered, certainly to a less extent, but, nevertheless, appreciably; and, as my table indicates, the lower the Reichert-Meissl number, the lower the maximum Polenske number, and, therefore, the greater the contrast between the increased Polenske number, due to the addition of cocoanut oil, compared with the corresponding maximum number for pure butter. As an example: In Table II. the Reichert-Meissl number 31 c.c., corresponding to a maximum Polenske number of 3·3 c.c., the addition of 10 per cent. cocoanut oil

(Reichert-Meissl number, 7·8; Polenske number, 17·6) to a butter giving these figures, would give a mixture yielding theoretically the following results:

Reichert-Meissl number = 28·6; Polenske number = 4·73.

If this Polenske number, 4·73, be compared with the maximum number for a Reichert-Meissl number, 28 to 29 (Table II.), which is 2·9 c.c., then we have the maximum Polenske number, 2·9 c.c., compared with 4·73; and $4·73 - 2·9 \times 10 = 18·3$ per cent. cocoanut oil.

Employing the maximum Polenske figure (Table II.) as a basis of calculation, I find that the mixtures of butter and cocoanut oil containing 5, 10, and 15 per cent. of the latter ingredient, show by this method 2, 7, and 18 per cent. cocoanut oil respectively, results, whilst laying no claim to scientific accuracy, are nevertheless of considerable value.

In addition, the simplicity of the method, and the fact that by one operation both the Polenske and Reichert-Meissl numbers are determined, and that duplicate estimations—if the conditions specified by Polenske are strictly adhered to—agree very closely, are strong recommendations for the adoption of this method in the routine examination of butter samples.

In conclusion, I must point out that the employment of cocoanut-oil cake as a feeding-stuff, and the influence that such a food-stuff is likely to exert on the analytical data of butter samples, must be considered.

Juckenack and Pasternack* in a recent paper give analytical data obtained by the analysis of butter derived from cows fed on cocoanut-oil cake. Whilst these figures were indicative of the addition of cocoanut oil, the absence of phytosterin was proved by the melting-point of the acetate compound being 113° C.

The authors conclude, from their analysis of many thousands of samples, that samples giving such abnormal figures are extremely rare; still, the possibilities of such figures being obtained with a perfectly genuine butter show the necessity, if the Polenske number is indicative of the addition of cocoanut oil to the extent of 10 per cent. or less, of corroborating the presence of cocoanut oil by the phytosterol acetate test.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

EXAMINATIONS IN CHEMICAL TECHNOLOGY.

THE first Examinations in Chemical Technology were held at the Institute from October 16 to 18, 1906. Three Candidates presented themselves on this occasion for the Examination, one of whom (Mr. James McLeod, F.I.C., Chief Chemist to the Glasgow Corporation Gas Trust) satisfied the Board.

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* *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1906, xi., 156.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Occurrence of Tannic Acid in Natural Wines. Jul. Mayer. (*Chem. Ztg.*, 1906, xxx., 898.)—The author states that in his experience all natural wines contain tannic acid. A considerable portion of this may be removed by treatment with gelatine, but never the whole. A wine free from tannic acid is to be regarded as a spurious article.

A. G. L.

Java Olive Oil. K. Wedemeyer. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 210-212.)—The Java olive is the seed of one of the *Sterculiaceæ*, and consists of an outer, parchment-like husk enclosing a hard shell, which contains the fleshy part of the seed. The shell yields about 10 per cent. of a yellow, butter-like fat, and the fleshy part 46·6 per cent. of a bright yellow oil, whilst the whole seed (husk, shell, and flesh) yields 30·3 per cent. of oil. This oil from the whole seeds is similar in appearance to ordinary olive oil, has a slightly rancid smell, but an agreeable taste. The following are its chemical and physical constants :

Specific gravity at 15° C.	0·9260
Refractive index at 40° C.	1·4654
Iodine value	76·6
Saponification value	187·9
Free acid	2·6
Acetyl value	23·5
Reichert-Meissl value	0·8

When heated to a temperature of 240° to 245° C., the oil is converted quite suddenly into a gum-like substance, sufficient heat being evolved during the change to carbonize the mass, unless the latter be cooled immediately. This gum-like substance is insoluble in all known solvents, and does not harden when exposed to air and light. The fatty acids separated from the oil behave similarly; when dried at a moderate temperature they become viscous, whilst prolonged heating converts them into a gummy substance.

W. P. S.

The Phosphorus Content of White of Egg. K. Kaas. (*Monatsh. Chem.*, 1906, xxvii., 403-409.)—The quantity of phosphorus contained in white of egg, as shown by the results of analyses given by the author, varies considerably, and appears to depend on the age of the egg. The albumin of a perfectly fresh egg yielded 0·155 per cent. of phosphorus, whilst that obtained from an egg (laid the next day by the same hen) which had been kept for one month contained 0·228 per cent. Possibly the white of egg withdraws phosphorus compounds gradually from the yolk. As much as 3·06 per cent. of phosphorus was found in crystallized albumin prepared by the Hammersten or Hofman-Pinkus process.

W. P. S.

Egg-Yolk preserved with Fluorides. Frabot. (*Ann. de Chim. Anal.*, 1906, vol. 11, p. 330.)—It is stated that for some time past small quantities of fluorides have been found in commercial samples of egg-yolk and broken eggs sold for food purposes. The presence of fluorine was readily detected by drying the samples with the addition of sodium carbonate, igniting the residue, and testing the alkaline ash. It was proved that the normal white or yellow of egg did not contain any natural compound of fluorine. C. A. M.

Determination of Crude Fibre in Cocoa Powders. H. Matthes and F. Müller. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 159, 160.)—The authors employ a slight modification of König's method (ANALYST, 1898, p. 47) for the determination of crude fibre. Five grams of the fat-free cocoa are digested with sulphuric acid and glycerol as described in the original process. After cooling, the mixture is diluted with water and allowed to settle over-night. The clear solution is then decanted on to an asbestos filter, and the residual liquid is mixed with its own volume (about 80 c.c.) of alcohol, and boiled for five minutes before bringing the whole on to the filter. The residue of crude fibre is washed successively with hot water and hot alcohol, then with ether, the asbestos and the residue being next transferred to a platinum basin, dried and weighed. The contents of the basin are finally incinerated, re-weighed, and the weight subtracted from the first weight. The difference gives the amount of crude fibre in the weight of the sample taken. The results obtained by this process are generally from 3 to 4 per cent. lower than those obtained by König's original process; in some cases the difference amounts to over 5 per cent. At the same time the results are about 0·5 per cent. lower than those yielded by Henneberg's process.

König, in a note on the above paper, considers that the slight modification introduced into his method by Matthes and Müller does not account for the wide difference in the results given by the two methods, and is of opinion that it is due to some other cause. W. P. S.

Determination of Crude Fibre in Cocoa. W. Ludwig. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 153-159.)—A determination of the amount of crude fibre yielded by a sample of cocoa is recommended as a means of detecting the presence or absence of added husk in the same: two grams of the finely powdered, fat-free cocoa are boiled for fifteen minutes with 20 c.c. of 15 per cent. sodium hydroxide solution and 60 c.c. of water. The mixture is then neutralized with hydrochloric acid, a further quantity of 10 c.c. of hydrochloric acid (specific gravity 1·125) is added, and the whole heated for two hours in a boiling water-bath. The insoluble portion is now collected on a filter, washed with hot water, and then rinsed back into the flask with about 70 c.c. of water; 1 gram of anhydrous sodium carbonate is added, the mixture is boiled for fifteen minutes, and filtered. The residue is again rinsed back into the flask with 100 c.c. of water and boiled, with the addition of 5 c.c. of concentrated hydrochloric acid for fifteen minutes, and again filtered. This treatment with sodium carbonate and hydrochloric acid is repeated, the residue obtained finally being collected on a weighed filter, washed with water, alcohol, and ether, dried and weighed. The filter and its contents are then ignited, and any ash obtained is subtracted from the total weight. Six samples of cocoa powder examined by the author gave from 4·98 to 5·96 per cent. of crude fibre calculated on the fat-free substance, the average amount being 5·60 per cent. These samples contained from 25·05 to 27·92 per cent. of fat. A sample of cocoa husk, containing 3·08 per cent. of fat, yielded 14·47 per cent. of crude fibre after the fat had been removed. A mixture consisting of equal parts of the above-mentioned six cocoas was then made, and to portions of the mixture varying percentages of husk were added. Determinations of the crude fibre in these mixtures gave the following results:

Composition of Mixture.	Fat.	Crude Fibre in Fat-free Powder.	
		Found.	Calculated.
	Per Cent.	Per Cent.	Per Cent.
Cocoa	25·78	5·50	5·60
,, 5 per cent. husk	24·37	6·03	6·16
,, 10 per cent. husk	24·00	6·39	6·71
,, 20 per cent. husk	21·44	8·06	7·79
,, 40 per cent. husk	16·62	10·18	9·74
,, 60 per cent. husk	12·04	11·72	11·48
,, 80 per cent. husk	8·26	13·28	13·04

W. P. S.

The Pentosan Content of Cocoa and its Value in the Detection of Husk in Cocoa Powders. H. Lührig and A. Segin. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 161-164.)—Sixteen samples of shelled cocoa beans examined by the authors yielded from 2·51 to 4·58 (average, 3·34) per cent. of pentosan, calculated on the dry, fat-free substance; whilst eight samples of husk gave from 7·59 to 11·23 (average, 9·26) per cent. of pentosan. From these results it is concluded that the determination is of no great value for the detection of husk in cocoa powder, as considerable quantities of husk may be added to cocoas containing little pentosan without the above limits being exceeded. Tollen's process (ANALYST, 1899, p. 179) was employed for the determinations. W. P. S.

Determination of Ergot in Flour. R. Bernhart. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 321-340.)—In the method described the flour is treated successively with hydrochloric acid, carbon tetrachloride, and ammoniacal copper solution, and the insoluble residue weighed. One hundred grams of the flour are boiled with about 500 c.c. of 2 per cent. hydrochloric acid until the starch is completely inverted, as is shown by testing a drop of the solution with iodine. After being allowed to settle, the clear liquid is poured through a dried and weighed silk filter on to which the residue is also brought. This residue is then extracted with carbon tetrachloride to remove fat. The residue is next removed from the filter by the aid of a spatula and placed in a beaker, where it is treated with ammoniacal copper solution. The latter must be freshly prepared and of sufficient strength to dissolve cotton-wool readily. When the cellulose present in the residue is dissolved, the mixture is diluted with ten times its volume of water, the residue is again collected on the filter, washed, dried at 100° C., and weighed. The difference between the weight of residue obtained from 100 grams of flour containing ergot and the weight of residue yielded by the same amount of flour free from ergot is multiplied by the factor 8·333 to give the percentage of ergot in the sample under examination. The method gives approximately accurate results.

If corn-cockle (*Agrostemma githago*) be present the ergot will be over-estimated by the above process. In this case the sample is boiled for two hours with 5 per cent. hydrochloric acid, filtered, the residue is boiled for one hour with 3 per cent. sodium hydroxide solution, and the insoluble portion separated and dissolved in concentrated hydrochloric acid. The solution is diluted with fifty times its volume of ice-water and kept in a cold place for several days. The chitine gradually separates out, forming a voluminous white precipitate, which is collected on an asbestos filter, dried and weighed. The weight of the precipitate multiplied by 43·38 gives the quantity of ergot in the portion of the sample taken for the analysis.

W. P. S.

The Quantity of Solanine in Potatoes. M. Wintgen. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 113-123.)—The amount of solanine existing in potatoes varies considerably, different kinds of potatoes yielding widely varying quantities of the glucoside. Twenty-eight samples of sound potatoes, including eleven varieties, yielded from 0·0172 to 0·1059 gram of solanine per kilo; generally, the quantities found were much less than those recorded by previous investigators. Storing the potatoes for a long time, even if germination takes place, does not increase the solanine content, and diseased potatoes were not found to contain more of the glucoside than sound ones. The formation of solanine by bacteria, as observed by von Weil, could not be established. W. P. S.

The Ash of Pimento, Cloves, and Cardamoms. R. Thamm. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 168-172.)—Continuing his work on the analysis of spices (*cf.* ANALYST, this volume, p. 231), the author now gives the results of the examination of various samples of pimento, cloves, and cardamoms. Nine samples of pimento gave the following results, the figures representing percentages on the dry, sand-free substance:

	Ash.			Alkalinity of Ash: c.c. N Acid per 100 Grams.		
	Total.	Water Soluble.	Water Insoluble.	Total.	Water Soluble.	Water Insoluble.
Average ...	4·56	2·58	1·98	68·9	25·3	43·5
Highest ...	4·86	2·80	2·31	74·6	27·9	49·8
Lowest ...	4·25	2·25	1·58	63·0	23·3	37·9

Six samples of cloves yielded:

Average ...	6·54	3·66	2·88	97·5	37·7	59·8
Highest ...	6·82	3·76	3·19	104·9	39·7	65·6
Lowest ...	6·29	3·56	2·62	94·4	32·9	56·2

Two samples of cardamoms (whole fruit) ground by the author gave:

1	7·08	4·46	2·62	67·2	11·2	56·0
2	3·33	1·10	2·23	50·5	6·6	43·9

W. P. S.

Oil of Sandalwood. A. R. L. Dohme and H. Englehardt. (*Amer. Druggist;* through *Pharm. Journ.*, 1906, vol. lxxvii., p. 325.)—From the results of the examination of thirteen samples of the oil distilled from the wood in the authors' laboratory, it is concluded that the specific gravity of the oil should lie between the limits 0·965 and 0·980 at 25° C., that the quantity of santalol should be from 91 to 92 per cent., and that the oil should be perfectly soluble in, at most, five volumes of 70 per cent. alcohol at a temperature of 25° C. The yield of oil obtained from the different parcels of wood varied from 3·95 to 6·18 per cent. W. P. S.

ORGANIC ANALYSIS.

A New Method of Detecting and Determining Methyl Alcohol. E. Voisenet. (*Bull. Soc. Chim.*, 1906, xxxv., 748-760.)—The method is based upon the oxidation of the methyl alcohol and detection of the methylal produced by the violet coloration which it gives with proteids and nitrous compounds (ANALYST, xxxi., 30). The yellow and violet colorations given by acetals in this test are really due to the acetaldehyde and formaldehyde respectively formed in the decomposition of the acetals by hydrochloric acid. For the detection of traces of methyl alcohol in ethyl alcohol it is necessary to separate by distillation the oxidation products of the latter (acetaldehyde and ethylal), so as to obtain a fraction in which the violet coloration will not be masked by yellow: A volume of the alcohol corresponding to 10 c.c. of absolute alcohol is diluted to 50 c.c. and treated with 5 grams of powdered potassium bichromate and 30 c.c. of sulphuric acid (1 : 5), and the mixture shaken and allowed to stand for an hour at the ordinary temperature. The products of the oxidation are now distilled in a 125 c.c. flask with a short neck, connected with a condenser 4 mm. in diameter and 30 cm. in length, the distillation being conducted so as to collect the first 30 c.c. of distillate in an hour. If the oxidation and distillation are properly carried out, the whole of the acetaldehyde derived from the 10 c.c. of absolute alcohol should be present in this first fraction of 30 c.c. On now continuing the distillation, the distillate will cease to give the yellow colour in the test, and the next 20 c.c. passing over should contain the methylal (boiling-point 42° C.), whilst the final fraction will contain the ethylal (boiling-point 104° C.). The method is stated to be capable of detecting 1 part of methyl alcohol in 20,000 parts of ethyl alcohol without any preliminary concentration. The method described above should be followed when the proportion of methyl alcohol lies between 1 : 10 and 1 : 1,000. In the case of a greater proportion than 1 : 10, the volume of alcohol (corresponding to 10 c.c. of absolute alcohol) is diluted to 100 c.c., and the liquid treated with 10 grams of potassium bichromate and 60 c.c. of sulphuric acid (1 : 5). The products of the oxidation are distilled very slowly, the first 50 c.c. being rejected and the next 20 c.c. collected. For smaller proportions than 1 : 1,000, the volume of alcohol is diluted to 25 c.c., and treated in a 75-c.c. flask with 2 grams of potassium bichromate and 12 c.c. of the sulphuric acid. The first 15 c.c. of distillate (obtained in thirty minutes) are rejected, and the test for methylal applied to the next 10 c.c. When only traces (1 : 10,000 or 20,000) are present, it is advisable to reduce the quantity of albumin and nitrous acid used in the test.

A colorimetric determination may be made by this method, the colour scale for comparison being prepared as follows: 10 c.c. of absolute alcohol containing 1 part in 50 of methyl alcohol are oxidized and distilled as above described, and the second fraction of 20 c.c. collected. To 4 c.c. of this fraction are added 1 c.c. of a 10 per cent. solution of albumin and 15 c.c. of hydrochloric acid containing nitrous nitrogen, this giving the maximum in scale. The second tube will contain 2 c.c. of the distillate, 2 c.c. of water and the reagents, and the colour will represent 1 : 100, and so on. The different tubes are kept for about fifteen minutes in water at 50° C., after which the colour in each will have attained its maximum. The colour scale, once prepared, can be kept without alteration for several weeks. C. A. M.

Nature of the Starch-Iodine Blue Compound. M. Padoa and B. Savarè. (*Gazzetta*, xxxvi., 310; through *Nature*, 1906, p. 473, and *Pharm. Journ.*, 1906, vol. lxxvii., p. 305.)—In order to elucidate the nature of the blue compound formed by the action of iodine on starch-paste, the authors have investigated the change in the electrical conductivity of a solution of iodine in potassium iodide caused by the addition of starch in known quantities. The conclusion drawn from the experiments is that the blue substance is an additive compound of iodine, starch, and potassium iodide (or hydrogen iodide) containing the two former constituents in the ratio $I : C_6H_{10}O_5 = 1 : 4$. Whilst this opinion supports that of Mylius, recorded some twenty years ago, it is opposed to the more recent view of Küster that the blue substance is not a definite compound, but is formed as a result of absorption by the colloid starch. Küster's contention received support quite recently from the work of Biltz, who showed that basic lanthanum acetate, which resembles starch in its colloidal nature, also produces with iodine an intensely blue substance similar in all respects to that formed from starch. In this case there is no reason to consider the substance as a definite chemical compound. W. P. S.

A New Reagent for the Detection of Mechanical Wood Pulp. A. Bergé. (*Bull. Soc. Chim. Belg.*, 1906, xx., 158, 159.)—An acid solution of *p*-nitraniline is stated to keep indefinitely, and to be a very sensitive reagent for mechanical wood pulp. It is prepared by dissolving 0·2 gram of *p*-nitraniline in 20 grams of sulphuric acid (66° B.) and 80 grams of water. Paper containing wood pulp is coloured by this solution a shade between orange and brick-red. The coloration is clearly distinguishable on isolated fibres, and the particles of wood can easily be counted under the microscope. A great advantage of this reagent is that it does not give any coloration with unbleached chemical cellulose, unless the boiling has been defective. The orange-red colour can be distinguished in artificial light. C. A. M.

Investigation of Celluloid. W. Will. (*Zeits. angew. Chem.*, 1906, xix., 1377.)—The author finds that properly made celluloid is a comparatively inert substance, which catches fire only on contact with a flame, and not by friction, concussion, an electric spark, or heating to above 100° C. Celluloid of inferior manufacture, however, will catch fire much more easily—*e.g.*, on being kept in the neighbourhood of the source of warmth of a heated room. Such celluloid has also a low temperature of decomposition. Celluloid itself burns quietly without exploding, but both the dust produced in working it and the gases yielded by its decomposition or combustion in an inadequate supply of air may give rise to explosions. The latter gases may also produce poisonous effects from their content of carbon monoxide, nitric oxide, or even hydrogen cyanide.

The temperature of decomposition of celluloid was determined by placing 0·1 gram of the sample in small pieces in a stout-walled, loosely corked test-tube. This was then plunged into an oil-bath previously heated to 100° C., after which the temperature was raised at the rate of 5° per minute until the celluloid decomposed. For the drying of the samples a special doubled-walled oven was used, in which the samples were placed on a tray, which was rotated so as to hasten the drying, the temperature being kept at 110° C. A. G. L.

On the Analysis of Celluloid. Hugo Dubovitz. (*Chem. Ztg.*, 1906, xxx., 936.)—Two grams of the finely-divided sample are shaken with 100 c.c. of acetone in a 150-c.c. flask until everything except the mineral matter has dissolved. The flask is then made up to the 150-c.c. mark with acetone, and 100 c.c. of the mixed contents transferred to a 100-c.c. flask. To the residual 50 c.c. of solution, 25 of an aqueous 8 per cent. ammonium chloride solution are added, and the whole well shaken, with simultaneous cooling in running water. This treatment yields the nitro-compounds in a coagulated, easily-filtered form, which is not the case if water only is used to separate them from the acetone solution. The precipitate is filtered off on a Gooch crucible, first washed with a mixture of 1 part of acetone and 1 part of the ammonium chloride solution to remove the camphor, then with absolute alcohol to remove ammonium chloride, finally dried and weighed. The ash is then determined by adding a little paraffin-wax, and melting it at a temperature below 100° C., after which the crucible is heated cautiously and finally more strongly. The addition of the paraffin-wax prevents detonation. Camphor is determined by subtracting the weight of nitro-compounds plus ash obtained from the weight of celluloid present in the 50 c.c. of solution.

The other 100 c.c. of solution are used in judging the quality of the nitro-compounds present. After precipitating the latter as above with ammonium chloride solution, the precipitate is shaken for thirty minutes in a stoppered 150-c.c. cylinder with 50 c.c. of absolute alcohol; 100 c.c. of ether are then added, and the whole allowed to stand, with occasional shaking, for twelve hours; 50 c.c. of the clear liquid are then evaporated to dryness in a platinum crucible, giving the soluble nitro-cellulose. As a check, soluble nitro-cellulose may also be determined in another part of the solution by precipitating with chloroform, filtering through a linen filter, again dissolving in alcohol and ether, reprecipitating with chloroform, drying at 60° C., and weighing. On another portion of the solution the total N_2O_5 is determined as usual with the nitrometer.

The temperature of decomposition is determined by placing the celluloid, cut into pieces, but not filed, in a test-tube in an oil-bath heated to 100° C., and slowly increasing the temperature. The temperature of decomposition rises markedly with the ash content, as shown in the following table:

Sample.	Camphor.	Total Nitro-cellulose.	Soluble Nitro-cellulose.	NO$_3$.	NO$_3$ in the Nitro-cellulose.	Ash.	Temperature of Decomposition.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	° C.
1	21·98	68·1	63·77	24·0	35·26	9·86	136
2	35·0	64·9	59·1	23·96	36·78	0	103
3	19·6	66·7	—	—	—	13·63	161
4	18·3	71·3	—	—	—	10·46	131
5	32·2	67·7	—	—	—	0	115

A. G. L.

Determination of Picric Acid. E. Feder. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 216.)—Owing to its acid properties, trinitrophenol is capable of liberating iodine from an aqueous solution of potassium iodide and iodate. The trinitrophenol acts as a mono-basic acid, and the quantity of iodine set free is proportional to the amount of trinitrophenol added. The titration of the liberated iodine with thiosulphate solution, using starch solution as indicator, is quite simple, and the change of colour at the end of the titration is sharp. One c.c. of $\frac{N}{10}$ thiosulphate solution corresponds with 0·0229 gram of trinitrophenol. The latter may also be determined in solutions of its salts by acidifying the same with hydrochloric acid, extracting the mixture with benzene, and evaporating the extract. The residue of trinitrophenol is dissolved in water, and its quantity determined as described above. Aqueous solutions of trinitrophenol may be titrated directly with alkali, using phenolphthalein as indicator, provided that a sufficient quantity of the latter be added. W. P. S.

The Use of Chromed Hide Powder in the Analysis of Tannins. E. Nihoul. (*Bull. Soc. Chim. Belg.*, 1906, xx., 236-240.)—The author has made comparative experiments with samples of chromed hide powder containing 0·6 to 1 per cent. and 4 to 5 per cent. of chromic oxide respectively and a sample of ordinary hide powder a year old, the determinations being made by the standard method. He found that the chromed powder yielded to distilled water only about 2·0 mgms. as against 2·1 mgms. from old ordinary hide powder; and from this concludes that the latter undergoes profound alterations on keeping, whatever precautions may be used to preserve it. In further experiments, in which an extract of quebracho, prepared under the standard conditions, was used with 5·5 grams of the different powders, it was found that the hide powder that was only slightly chromed gave an exact result in the determination of the non-tannin matter, whereas the ordinary old hide powder gave a figure more than twice too high, although it was remarkable that the quebracho solution had dissolved less of the soluble substances in this powder than had the distilled water. The results obtained with the highly-chromed powders were also incorrect, and it was found necessary to use at least 12 grams of them to obtain figures in any degree concordant with those yielded with the lightly-chromed powder. Hence the author prefers the use of 5·5 grams of the latter, provided, of course, that the proportion of chromic oxide is sufficient to preserve the hide powder.

C. A. M.

The Quantitative Determination of Carbon Dioxide and of Carbon. John McFarlane and Arnold W. Gregory. (*Chem. News*, 1906, vol. 94, p. 133.) —When small quantities (0·01 gram) of carbon dioxide are to be determined, the error caused by varying condensations of moisture, etc., on the potash bulbs usually employed is considerable, and the authors consequently prefer in such cases to absorb the gas in standard barium hydrate solution, filtering off the barium carbonate formed, and converting it into barium sulphate, which is specially suited to this purpose on account of its relatively high molecular weight. As an additional check, the filtrate from the barium carbonate is titrated with standard acid. In order to avoid the action of the carbon dioxide in the air during the filtration of the baryta solution, the authors make use of the apparatus shown in Fig. 1, which combines the functions of an absorbing and filtering vessel. The wide filter-tube (A) is closed during absorption by a piece of rubber tubing and a clip. The filtering medium in it consists of a piece of glass wool covered by a layer of paper or asbestos pulp, kept in place by placing on it a little broken glass. A measured volume of the baryta solution is introduced through B into A after the apparatus has been swept out with pure air; the gas is then allowed to enter through B, and drives some of the liquid up the spiral tube (C) into the second bulb, the absorption thus being made very efficient. The barium carbonate obtained after washing with boiled-out water is dissolved out of the apparatus with dilute hydrochloric acid, and converted into barium sulphate as usual.

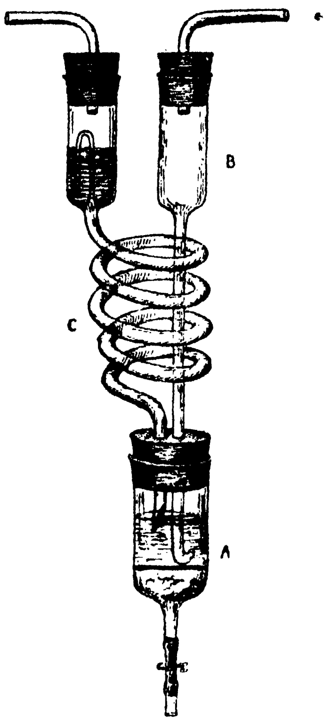


FIG. 1.

If the quantity of barium carbonate is large, filtration is very tedious, on account of the gelatinous nature of the precipitate, and the authors then recommend the use of the apparatus shown in Fig. 2, in which an ammoniacal solution of barium chloride constitutes the absorbing medium. This solution contains 50 grams of barium chloride and 250 c.c. of 0·880 ammonia per litre. The solution is boiled just before use, and filtered hot through the filter-tube (C), provided with a tap, into the first of the two flasks (A), the connecting-tubes being of such a length that a part of the liquid is afterwards driven over into the second flask (B) by the gas itself. At the end of the absorption the flask (A) is

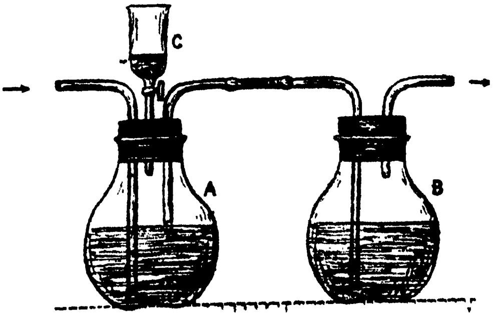


FIG. 2.

heated until it boils, the steam issuing from it heating (B) sufficiently to precipitate all the barium carbonate in this flask. Both flasks are then allowed to cool, and are then only disconnected and their contents filtered. Practically no precipitation of barium carbonate takes place in cold ammoniacal solutions. The barium carbonate obtained is converted into barium sulphate, as before. The results obtained in this way are good. A. G. L.

The Determination of Tannin in Tanning Materials. Boudet. (*Bull. Soc. Chim.*, 1906, xxxv., 760-762.)—The method is a more exact and simpler modification of that of Jean, and depends on treating the solution of the tannin with an excess of iodine and titrating the uncombined excess by means of standard sodium thiosulphate solution. It has been found by numerous determinations that 1 gram of iodine combines with 1·137 gram of pure dry tannic acid. The reagent is prepared by dissolving 4 grams of pure dry iodine and 8 grams of potassium iodide in a litre; whilst the thiosulphate solution contains 7·81 grams per litre. Since 4 grams of iodine should saturate 4 × 1·137 grams of pure tannin, the latter quantity of the sample is dissolved in a litre of water, and 10 c.c. of the solution treated with 10 c.c. of the iodine solution, and allowed to stand for two hours. The excess of iodine is then titrated by means of the thiosulphate solution, and the number of c.c. of iodine solution, less the number of c.c. of thiosulphate solution, gives directly the percentage of pure tannin in the sample.

Gallic acid may be determined in the same way, 1 gram of iodine combining with 0·467 gram of that acid. Hence 4 × 0·467 = 1·87 grams of a commercial sample are taken for a determination. In the case of a mixture of tannic and gallic acids 4·54 grams are taken, and the two acids determined together. The tannin in the solution is then absorbed by means of hide powder and a second determination made of the gallic acid in the filtrate in terms of tannin, which can be corrected into gallic acid by multiplying by the factor 0·41. C. A. M.

Measurements of the Temperatures of Gelatinization and of Specific Gravities of some Glue Solutions. K. Winkelblech. (*Zeits. angew. Chem.*, 1906, xix., 1260.)—Constant results for the temperatures of gelatinization of glue solutions are obtained by vigorously agitating in a cold water-bath a considerable amount of the solution (400 c.c.) contained in a flask (of 500 c.c. capacity), and noting the point at which a thermometer placed in the glue solution remains stationary for some time. The flask must be well shaken during the cooling, especially if dilute (2 per cent.) solutions are being examined, as otherwise there is a possibility of supercooling.

Determinations of the specific gravity at various temperatures by weighing agreed well with results given by the hydrometer, after allowing for the small correction needed, on account of the drawing up of the opaque liquid around the hydrometer by surface tension. A table giving results obtained for some German glues is also given. A. G. L.

Characteristics of Roasted Copals and of Copals treated with Phenols or Naphthalene. C. Coffignier. (*Bull. Soc. Chim.*, 1906, xxxv., 762-767.)—When copals are heated in an autoclave with phenols or naphthalene at a temperature of 260° to 290° C., and at a pressure of 2 to 6 atmospheres, they are rendered, without loss, soluble in a mixture of linseed-oil and turpentine (French Pat. 334,300, October, 1904). The same result is obtained, though with considerable loss, on simply roasting the copals. In order to determine the effect of the two methods upon the general properties of copals, the author has made comparative experiments with Madagascar copal, and his chief results are as follows :

	Specific Gravity at 16° C.	Melting-point.	Acid Value.	Saponification Value.	Insoluble in			
					Alcohol.	Ether.	Chloroform.	Benzene.
		° C.			Per Cent.	Per Cent.	Per Cent.	Per Cent.
Natural Madagascar copal	1·0588	300	93	70·1	73·8	65·0	69·0	78·4
Madagascar copal, roasted	1·062	205	68·2	44·9	91·8	51·8	1·7	1·5
Madagascar copal rendered soluble with naphthalene	1·061	165	68	65·9	74·4	19·7	soluble	40·0

Roasted copal is very soluble in oil of turpentine, whereas copal treated with naphthalene is only slightly soluble (about 50 per cent.). On the other hand, copal treated by the naphthalene process is completely soluble in amyl acetate, whilst the roasted copal is only very slightly soluble. Whichever method is employed the acid value is invariably lowered. The specific gravity is somewhat increased, and the melting-point and saponification value greatly lowered. C. A. M.

Optical Analysis of Technological Gas Mixtures. F. Haber. (*Zeits. angew. Chem.*, 1906, xix., 1418.)—The author has worked out an entirely new method for the instantaneous examination of a number of gas mixtures, which depends on the comparison of the refractive index of the gas mixture with that of a known gas or mixture. In the figure a diagram of the apparatus used, made by Carl Zeiss, is shown. Light from a lamp (not shown) goes through a prism (I), through the objective of the telescope (Ob), to a double reflecting prism; thence through the actual gas prism and its envelope (P) to a mirror (Sp), and then back again, an

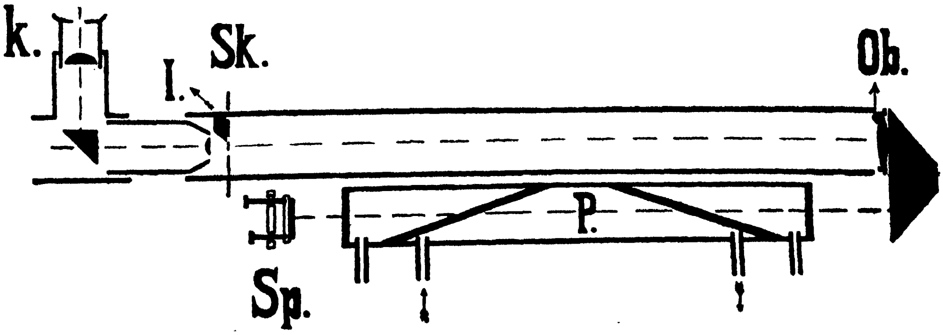


image of the prism (I) being formed on a real scale (Sk) placed close to (I). This image is viewed through a microscope, the eyepiece of which (k) is at right angles to the objective. If the prism below the eyepiece is removed, the light goes straight on to the left, where a camera or another eyepiece may be placed. The gas to be examined is sent either through the gas prism (P) or else through its envelope, the standard gas being sent through the other vessel. The size of the prism is 20 × 2 × 1 cm. It is made of glass plates cemented to a metal body. The relative refractive index of the two gases is given by the displacement of the image of the prism (I) along the scale.

The necessity for the use of a microscope becomes apparent when it is remembered that the refractive indices of gases vary only in the fourth or fifth decimal place. In fact, the gases so far examined fall into seven classes, the individual members of each of which cannot be compared with each other in this way, as their refractive indices are too close together. Each division of the scale is 0·02 mm. distant from the next, and corresponds to a difference in the refractive index of 0·0000015. The instrument is sensitive to about one-fifth of this value. As examples of the application of the method are given the determination of carbon dioxide in air, 1 per cent. of carbon dioxide corresponding to 1·1 scale divisions; the determination of hydrogen or methane in air, both about as accurate as that of carbon dioxide; and the determination of heavy hydrocarbons or benzene vapour in illuminating gas before and after enrichment, about ten times as sensitive as the foregoing. The author believes it possible to apply the instrument to a number of other simple technological determinations. A. G. L.

INORGANIC ANALYSIS.

The Colorimetric Determination of Small Quantities of Gold. Ralph Nelson Maxson. (*Zeits. Anorg. Chem.*, 1906, xlix., 172.)—Small quantities of gold may be readily determined colorimetrically by dissolving the gold in aqua regia, evaporating the solution to dryness on a water-bath, treating the residue with a small amount of an aqueous solution of acetylene, and comparing the colour of the red colloidal gold solution produced with the colour given by a known amount of a standard gold solution, a Penfield colorimeter being convenient for this purpose. Scrupulous care must be taken to ensure the absence of all electrolytes, which would coagulate the gold; treatment of the vessels used with steam is recommended. The addition of a few drops of ether also helps to prevent coagulation. If only very small quantities of gold are present, the volume of the solution used should not exceed a few c.c. Gentle warming of the residue before treating with the acetylene solution increases the rapidity of the reduction, but care must be taken that no gold is reduced to the metallic state at this point. As regards the accuracy of the results, the maximum error on quantities of gold of from 0·00080 to 0·00001 gram is about 10 per cent., and very often the error is much less than this. A. G. L.

The Separation of Iron and Manganese from Nickel and Cobalt. W. Funk. (*Zeit. anal. Chem.*, 1906, xlv., 562-570.)—In separating nickel and cobalt from iron and manganese by treatment of the precipitated sulphides with dilute acid, it is preferable to use formic acid instead of the usual hydrochloric or sulphuric acid. But even then the results are not so greatly improved that a single precipitation can yield an exact separation. The method, therefore, cannot be recommended in the case of iron, since the process must be repeated twice at least, and when considerable amounts are present, both the precipitate and filtrate must be treated. For the technical analysis of New Caledonian ores, it is best to separate the iron from the manganese, nickel, and cobalt by the acetate or other method, and then to separate the last three metals. For the separation of manganese from nickel and cobalt a single repetition of the process is sufficient, and it is not necessary to treat the precipitate again, since it will only contain traces of manganese. The following method is recommended: The solution, containing about 0·1 gram of nickel or cobalt in 200 c.c. and 2 per cent. of ammonium chloride or sulphate, is treated with ammonium sulphide in the smallest possible excess, and the precipitate allowed to subside until the liquid cools. A dilute (about normal strength) solution of formic acid is now added until the manganese sulphide has dissolved, and the solution contains about 0·5 to 1·5 per cent. of free acid. The liquid is next saturated in the cold with hydrogen sulphide, being meanwhile continually stirred, and the precipitate collected and washed with dilute hydrogen sulphide solution. The filtrate is concentrated slightly, and the treatment repeated in order to separate the small quantities of nickel and cobalt still remaining in solution. If iron has been separated first by the formate method, this treatment is preferable; but if the acetate method was used to separate the iron, it is best to precipitate the nickel and cobalt from an acetic acid solution by means of hydrogen sulphide. As regards accuracy, the two methods are equal.

C. A. M.

The Influence of Ammonia and Ammonium Salts on the Titration of Zinc by Schaffner's Method. A. Deckers. (*Bull. Soc. Chim. Belg.*, 1906, xx., 164-167.)—It is shown that in titrating zinc with sodium sulphide solution ammonium salts, whether in neutral or ammoniacal solution, decompose the sodium sulphide with the liberation of hydrogen sulphide, and that the amount of the latter produced is proportional to the quantity of ammonium salts present. They have a similar action upon freshly precipitated zinc sulphide, and the intensity of this reaction increases with the proportion of free ammonia. Hence, in order to obtain accurate results by Schaffner's method it is essential that the solution of the sample and the standard solution for the comparison should not only contain equal amounts of ammonia and ammonium salts, but also that the proportion of those salts should not be too high.

C. A. M.

The Iodometric Determination of Hydrogen Sulphide. O. Brunck. (*Zeit. anal. Chem.*, 1906, xlv., 541-551.)—Direct titration of hydrogen sulphide with iodine solution invariably yields too low results, whether the titration be done in neutral or in alkaline solution. Even when the determination is made very rapidly there is still a considerable loss. Correct results, however, can be obtained by the modification of Fresenius, in which the dilute solution of hydrogen sulphide is run into an insufficient amount of iodine solution, and the titration then completed with the same iodine solution. If the difference between the amount of iodine solution originally taken and the total amount consumed is considerable (more than 1 c.c. of $\frac{N}{100}$ iodine solution) the determination must be repeated, a larger amount of the original iodine solution being used. The method of back titration gives results in agreement with the gravimetric results, whether in a neutral or an alkaline solution. In the latter case the iodine solution must obviously be rendered acid before the titration with sodium thiosulphate solution. In the case of strong solutions of hydrogen sulphide, it is advisable to use sodium bicarbonate to prevent loss. Soluble sulphides can also be titrated by these methods, but it must be remembered that in practice solutions of alkali sulphides almost invariably contain thiosulphates.

C. A. M.

Remarks on the Usual Reactions for Nitric Acid. P. Soltsien. (*Pharm. Ztg.*, 1906, vol. 51, p. 765; through *Chem. Ztg.*, 1906, xxx. [*Rep.*], 309.)—Nitrous acid not only does not yield the reaction with diphenylamine given by nitric acid, chlorine, and other oxidizing agents, but actually hinders it. In presence of comparatively large amounts of nitrous acid small quantities of nitric acid may not give the reaction at all. Nitrous acid also interferes with the bromine reaction.

A. G. L.

Two New Colour Reactions of Nitric Acid. C. Reichard. (*Chem. Ztg.*, 1906, xxx., 790.)—*Arbutin* is coloured intensely yellow by concentrated nitric acid. To carry out the test on the free nitric acid, a drop of it may be added to a little solid arbutin on a white tile. If the acid is too weak, however, no colour is produced in this way. In that case a drop of strong sulphuric acid is added, or, better, the acid is converted into potassium or ammonium nitrate, which is evaporated to dryness, a little arbutin and a drop of sulphuric acid being then added. Nitrates are detected in a similar manner, as little as 0·00015 gram potassium nitrate giving a distinct colour. Hydrochloric acid may be used instead of sulphuric acid, but the mixture then requires warming, and the reaction is not so delicate. On absorbing the coloured solution in filter-paper and treating with alkalis, certain changes are produced. The colour given by hydrochloric acid is turned reddish-yellow by a 40 per cent. solution of potassium hydroxide, while the colour given by sulphuric acid is turned violet (in places only) by ammonia.

If a little *berberin hydrochlorate* is treated with a drop of nitric acid, and the mixture warmed till dry, a deep red-brown residue, which may be almost black in places, is obtained. In the presence of sulphuric acid the colour is produced in the cold with either nitric acid or a nitrate; 0·00002 gram nitric acid will give a deep colour. If hydrochloric acid is used instead of sulphuric the colour produced is the same, but is only obtained on warming. Acetic acid yields a violet-blue residue. A. G. L.

On the Standardization of Iodine and Thiosulphate Solutions. G. Bruhns. (*Zeit. Anorg. Chem.*, 1906, xlix., 277.)—Referring to Metzl's work on the same subject (ANALYST, 1906, xxxi., 96), the author shows that potassium bichromate is a perfectly suitable body for the standardization of thiosulphate solutions. The only precaution necessary to insure accurate results is that the mixture of bichromate, hydrochloric acid, and potassium iodide must be allowed to stand (in a closed vessel) for thirty minutes before the titration, in order to allow the reaction to complete itself. Still more convenient for the same purpose is the use of a standard solution of potassium permanganate, which, in the author's experience, can be preserved unchanged for a very long time, the advantage of permanganate over bichromate being that with the former the reaction, which results in the liberation of the iodine, is instantaneous.

A. G. L.

The Volumetric Determination of Sulphuric Acid by Means of Benzidine. E. Lemaire. (*Ann. de Chim. Anal.*, 1906, vol. 11, pp. 333-335.)—The methods based on the insolubility of benzidine sulphate in cold water have been studied by the author with special reference to the determination of sulphuric acid in hydrochloric acid. The difference between the results given by the methods of Muller (ANALYST, xxviii., 332) and of Raschig (ANALYST, xxviii., 333) hardly exceed 1 per cent., but a considerable error is introduced through the solubility of the benzidine sulphate in hydrochloric acid. To obviate this it is essential to carry out the titration only after complete neutralization of the acid, and the most accurate results are obtained by working at a temperature of 60° C. In the modification recommended by the author 5 c.c. of the hydrochloric acid under examination are mixed with 100 c.c. of water and a few drops of phenolphthalein solution, and titrated with standard sodium hydroxide solution, the result giving the total acidity. Ten c.c. of the benzidine hydrochloride solution (prepared as described by Raschig) are then added, and the flask shaken and allowed to stand for a few minutes. The precipitate is washed with the aid of the filter-pump and then returned to the flask, shaken with water at 60° C., and titrated with $\frac{N}{10}$ sodium hydroxide solution, with phenolphthalein as indicator. The result is calculated as hydrochloric acid and deducted from the amount of total acid previously determined. The benzidine method has been used with success by von Knorre (ANALYST, xxx., 170), in the determination of sulphur in crude pyrites, and the author has tried it upon the residue from roasted pyrites; but in this case the proportion of sulphur to iron is so small that considerable quantities of the substance are required, and the method therefore ceases to be practical.

C. A. M.

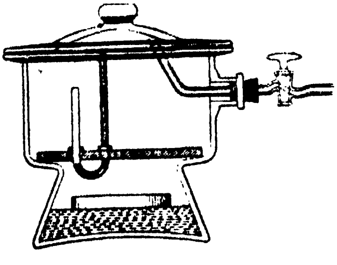
Causes of Error in the Determination of Phosphoric Acid by the 'Citro-Mechanical' Method. E. Guerry and E. Toussaint. (*Bull. Soc. Chim. Belg.*, 1906, xx., 167-203.)—The presence of silica is a great source of error in the determination of phosphoric acid in phosphates and basic slags by the citric acid method, since it is liable to be precipitated simultaneously; and the authors' experiments show that where accurate results have been obtained hitherto the amount of silica has been small, or it has been kept in solution by the presence of more or less iron. Thus, on adding to the acid solution of a basic slag a certain quantity of an iron salt the phosphoric acid can be precipitated without the silica, although in all exact determinations the silica ought to be eliminated before the precipitation. For technical purposes the following modification is recommended: 2·5 grams of the sample are boiled for thirty minutes with 100 c.c. of water and 50 c.c. of *aqua regia*, and the liquid cooled and made up to 250 c.c. To 25 c.c. of this solution are then added 25 c.c. of a solution of ferric chloride (prepared by diluting 100 c.c. of a 2 per. cent. solution to 500 c.c.), 50 c.c. of water, 30 c.c. of ammonium citrate solution (containing 10 grams of citric acid) and 15 c.c. of ammonia solution (specific gravity 0·91), and the whole mechanically shaken for thirty minutes, and then treated with 50 c.c. of magnesia mixture, which is added drop by drop. After standing for an hour the supernatant liquid is decanted, and the precipitate washed on to the filter with a 10 per cent. solution of ammonia containing 10 per cent. of the citrate solution. It is next washed on the filter with this liquid once, and finally with dilute ammonia solution, as in the ordinary method. The results thus obtained agree well with those given by the molybdate method.

Sulphuric Acid Method.—A generally applicable method for the determination of phosphoric acid is to treat the material first of all with hot concentrated sulphuric acid, which expels fluorine and renders the silica insoluble. 2·5 grams of the substance are heated with 10 c.c. of sulphuric acid in a Jena glass flask (or in a platinum basin when the sample contains much fluorine) with constant shaking until the appearance of fumes of sulphuric acid (which must not be confounded with those of silicon fluoride, which may be given off first). The contents of the flask are allowed to cool, cautiously diluted with water to 100 c.c., then mixed with 25 c.c. of *aqua regia*, boiled for ten minutes on a sand-bath, cooled, made up to a definite volume, and filtered. From 5 to 10 c.c. of the ferric chloride solution are added to 25 c.c. of the filtrate, in case the treatment with sulphuric acid was insufficient, and the liquid partially neutralized with ammonia, cooled, treated with 30 c.c. of citrate solution, 15 c.c. of ammonia solution, and 35 c.c. of magnesia mixture, and shaken mechanically for thirty minutes, after which the precipitate is allowed to stand for an hour, and then collected on a filter, washed, and ignited. C. A. M.

APPARATUS.

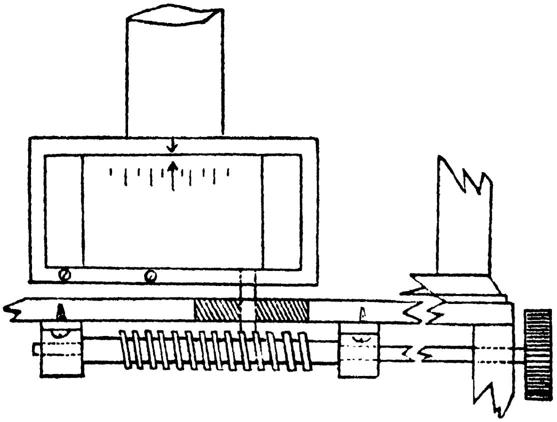
The Production of a High Vacuum in the Scheibler Desiccator.

H. C. Gore. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 834; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 294.)—The Scheibler desiccator is provided with an exit tube terminating just below the lid, and with a manometer, as shown in the figure. Two wash-bottles are inserted between the pump and the desiccator, one empty, to receive any water sucked back, the other containing a little water, by means of which the passage of the air from the desiccator can be watched. The desiccator is charged with about 200 c.c. of concentrated sulphuric acid, and also contains a 10-cm. Petri dish filled with ether. As soon as the pump is started the ether evaporates; when no more bubbles pass through the wash-bottle, the tap of the desiccator is closed, and the latter allowed to stand, or agitated gently. By the latter procedure

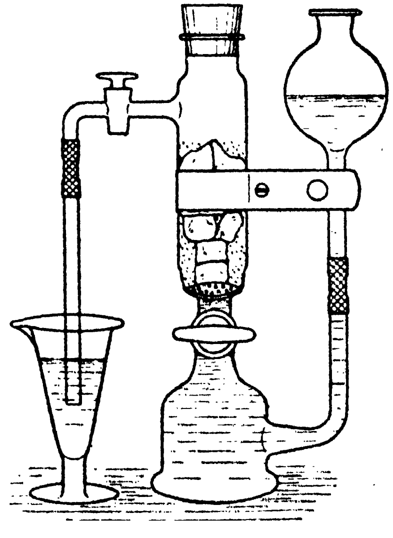


especially a very high degree of exhaustion (0·1 cm. or less) is rapidly obtained. When substances rich in water—*e.g.*, potato starch—are to be dried, some caution appears to be necessary to prevent the water from evaporating so rapidly as to throw the substance about the desiccator. A. G. L.

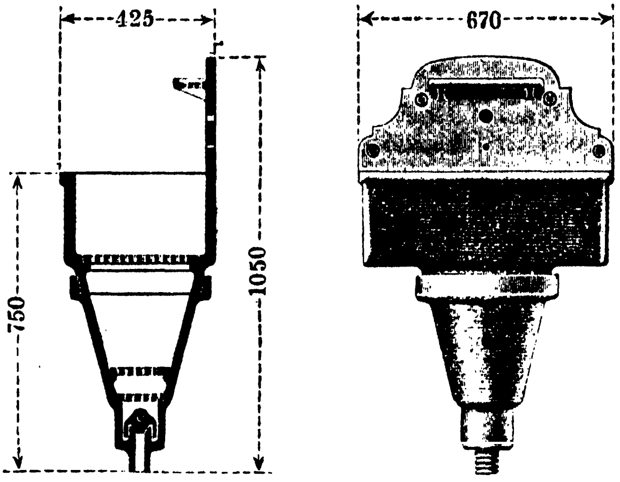
New Zero Adjustment for Chemical Balances. J. McDowall. (*Chem. News*, 1906, vol. xciv., p. 104.)—The ordinary pointer scale of the balance is replaced by a brass frame, in which a scale slides in grooves, as shown in the diagram. At the right-hand bottom corner of the scale is a projection which extends through the base of the balance case, and engages the screw-thread fitted below the base, and operated by the milled head shown on the right. If, on releasing the balance, the pointer swings, say, a few divisions further to the right than to the left, the scale is moved in the desired direction by means of the milled head until adjustment is obtained. In cases where the balance is completely out of adjustment, the usual counterpoising screws are turned to bring the pointer roughly back to the centre of the scale when the balance is released, the exact zero being then obtained by the above-mentioned device. W. P. S.



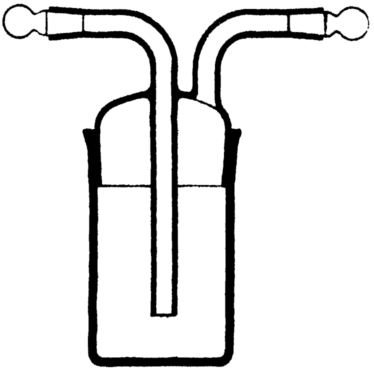
Improved Mayer's Apparatus for the Evolution of Chlorine. (*Zeit.* xii., 221-223, *Untersuch. Nahr. Genussm.*, 1906).—In clearing vegetable tissue for microscopical examination, chlorine evolved from hydrochloric acid and cubes of calcium hypochlorite in Mayer's apparatus is frequently used. Some disadvantages are attached to this apparatus, which have been remedied by slight alterations in its form. These consist of the addition of two glass taps, and doing away with the revolving glass stopper with a side aperture, by means of which the flow of acid was regulated. This, in the new form, is replaced by a rubber stopper, which does not bind with the hypochlorite, and the flow of acid is regulated by the upper tap. The closure of the lower tap effectually seals off the liquid from the solid contents after the liquid has been driven up into the side-tube. W. P. S.



A New Laboratory Sink. Heinrich Gockel. (*Chem. Ztg.*, 1906, xxx., 755.)—The novelty of the sink shown in the figure—which may be obtained from the firms, Dr. Heinrich Göckel and Deutsche Ton - und Steinzeugwerke, both of Berlin—consists in the lower funnel - shaped part. This enables long vessels, such as burettes and cylinders, to be readily washed under the tap in a vertical position. The exit-pipe of the sink may be closed by means of a stopper attached to a chain, and several removable perforated supports are provided, on which large vessels may be placed whilst they are being cooled in the filled sink. The sink will take flasks up to 5 litres and bottles up to 12 litres capacity in this way. A back-plate fitted with a small shelf, as shown, is also provided. The measurements are in mm. A. G. L.



Weighing Bottle. Leo F. Guttmann.—This is intended for the drying of a substance in an inert gas, such as hydrogen or nitrogen, in order to determine any volatile constituent it may contain. The bottle is made of light glass, and carries a stopper provided with gas inlet and outlet tubes. The little ground-in stoppers are exceedingly useful in keeping the bottle air-tight.



A similar weighing bottle to the one illustrated in this journal (this vol., p. 276) was invented, Mr. Guttmann says, by himself six years ago. A description of it is given in Jannasch's "Leitfaden der Gewichtsanalyse," Leipsic, 1902. It is manufactured and sold by several dealers in chemical apparatus. W. J. S.

A Temperature Regulator for Use with the Immersion Refractometer. F. Löwe. (*Chem. Zeit.*, 1906, xxx., 685, 686.)—The apparatus consists of a large vessel for holding water, and in which the beakers containing the solutions to be examined are successively immersed while the readings are being taken. A support, *T*, is provided for holding the beakers (Fig. 1) whilst the instrument is suspended from the rod, *B*, which is clamped to the sides of the water-vessel. The mirror, *S*, is fixed to the arm, *A*, and moved by the second rod seen in the diagram. In Fig. 2 is shown a larger form of apparatus, in which twelve or more beakers are fixed to the revolving portion, *SG*, and their contents simultaneously brought to the desired temperature before being examined by the instrument. This form of the apparatus is also provided with an outflow for the water and a specially devised distributor for the inflowing water, the latter being distributed through the vessel by a number of small pipes, the use of a stirrer being thus rendered unnecessary. W. P. S.

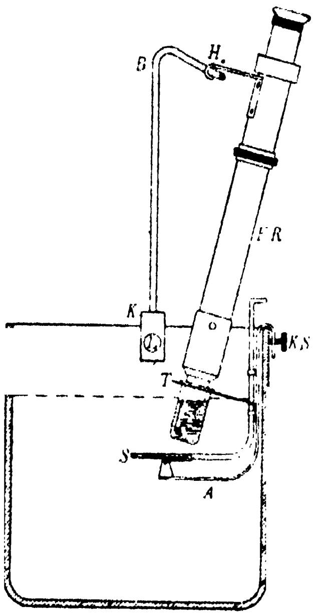


FIG. 1.

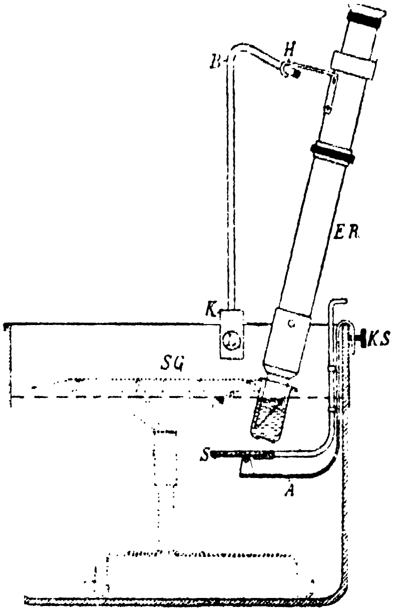


FIG. 2.

A Simple Method for Piercing Glass. P. N. Raikow. (*Chem. Ztg.*, 1906, xxx., 867.)—Holes of any desired size may be readily made in glass of any thickness up to 4 mm. by piercing the heated glass with a red-hot needle. The needle should be as pointed and as hot as possible; the glass, on the other hand, must not be warmed too much. The best procedure is to heat the needle in a Bunsen flame in such a position that its red-hot point penetrates just outside the Bunsen flame; the previously warmed glass is then pushed up against the point and gradually brought into the flame, the needle being at the same time twisted to and fro until it penetrates the softened glass. Before allowing the glass to cool, it should be well covered with soot to prevent cracking. Any number of holes may thus be made in a test-tube or flask, etc. For convenience in handling, the needle may be fused into a glass holder; an old thermometer forms the best holder for this purpose, as other glass usually cracks in contact with the steel needle. A. G. L.

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ADULTERATION OF WINE AND SPIRITS.

THE following extracts from a Bill to prohibit the use of certain foreign substances in wine, brandy, whisky, and spirits, passed by the Government of the Cape of Good Hope, will, in view of the recent agitation in this country regarding the sale of brandy, whisky, etc., be read with interest.

* * * * * *

5. Unless the context of the Act otherwise requires—" Wine " means the product solely of the alcoholic fermentation of the juice or must of fresh grapes. " Dry wine " means wine produced by complete fermentation of the sugar contained in the juice or must of the fresh grapes from which it is made. " Sweet wine " means wine containing sugar derived from the juice or must of the grapes from which it is made, and not produced from imported raisins. " Sparkling wine " means wine surcharged with carbonic acid gas, and to which cane-sugar and pure wine spirit may or may not have been added, and includes champagne. " Pure wine spirit " means the rectified distillate resulting from the distillation solely of wine or must. " Natural wine " or " pure natural wine " means the product solely of the alcoholic fermentation of the juice or must of fresh grapes without the addition of any foreign substance as hereinafter defined, before, during, or after the making of the same.

6. No person shall sell, advertise, offer, keep, expose, or deliver for sale, whether wholesale or retail, or exchange or authorize, direct or allow the sale, under the name, label, or designation of "natural wine," or "pure natural wine," or "natural dry wine," or "pure natural dry wine," or "natural sweet wine," or "pure natural sweet wine," or "sparkling wine," any substance except natural wine, natural dry wine, natural sweet wine, or sparkling wine, to which, before, during, or after the making of the same, no foreign substance has been added.

7. The term "foreign substance" used in this Act—(*a*) Shall include the following and such others as are afterwards made illegal, viz.: Ethers, essential oils, bitter almond, cherry laurel, flavouring substances, alkaloidal substances, compounds of barium, fluorine, magnesium, strontium, bismuth, arsenic, lead, zinc, aluminium, tin, copper, boron, derivatives of naphthol (abrastol, etc.), sulphuric acid, formalin or formaldehyde, salicylic acid, or other antiseptics (except sulphurous oxide as provided for hereinafter); glycerine, saccharine, dulcine, sucrovin, starch sugar, invert sugar, cane-sugar (except in the case of champagne), impure spirit containing more than 1 per mille of fusel oil, organic or mineral colouring matters, gums, and any mixture containing any of these substances; but (*b*) shall not include the following and such others as are afterwards made illegal: (1) Yeast or leaven. (2) Substances, such as isinglass, gelatin, eggs, albumin, Spanish clay, kaolin, or tannin for the purpose of clarification. (3) Common salt, provided that the total amount of chlorine in the wine, calculated as sodium chloride, does not exceed ½ gram per litre or 35 grains per gallon. (4) Sulphate of lime, metabisulphite of potassium or sulphurous oxide, provided that the total amount of sulphuric oxide, calculated as potassium sulphate, does not exceed 2 grams per litre or 140 grains per gallon. (5) Tartaric acid. (6) Natural products of grape-vine leaves or flowers. (7) Pure wine spirit, pure wine brandy, or spirit

distilled from sound wine at not less than 38° over proof, for the purpose of increasing the alcoholic strength to an extent not exceeding 28 per cent. of proof spirit or 16 per cent. of alcohol by volume in the case of dry wines, or 35 per cent. of proof spirit or 20 per cent. of alcohol by volume in the case of sherries, ports, and sweet wines, or 43 per cent. of proof spirit or 25 per cent. of alcohol in the case of imported wines, alcohol in either case being absolute alcohol of specific gravity 0·7938, and measured at the temperature of 60° of Fahrenheit's thermometer.

8. No person shall sell, etc., any wine to which has been added water, or cane-sugar in quantity exceeding 8 ounces per gallon, or salicylic acid in quantity exceeding 4 ounces per 127 gallons.

9. No person shall sell, advertise, offer, expose, or deliver for sale, whether wholesale or retail, or exchange, or authorize, direct or allow the sale of wine containing more than the following quantities of sulphurous oxide, namely : (*a*) In the case of dry wine, 14 grains of free and combined sulphurous oxide or 1½ grains of free sulphurous oxide per gallon ; and (*b*) in the case of other wines, 25 grains of free and combined sulphurous oxide or 2¼ grains of free sulphurous oxide per gallon.

10. Nothing in this Act shall be deemed to prohibit the sale of any fermented beverage made from fruit or sources other than fresh grapes, if such is correctly designated as to source and fruit it is made from.

11. Unfermented grape-juice to which there has been added any substance the addition of which to wine is prohibited, or hereafter shall be prohibited may, be sold, provided that nothing in this section shall be construed to place any restriction upon the manufacture or sale of temperance beverages or sacramental wine unless these contain more than 3 per cent. of proof spirit.

12. No person shall manufacture or sell under the name of champagne sparkling wine in which the excess of carbonic acid gas arises from direct admixture of the same.

13. The flavouring of wine other than natural wine and the colouring thereof by means of pure caramel shall not be deemed to be an offence.

14. Brandy, Whisky, and Liqueur.

"Brandy" means the distillate resulting from the distillation solely of (*a*) wine or must ; (*b*) must and grape-husks ; (*c*) grape-husks and water. "Pure wine brandy" means the unrectified distillate resulting from the distillation solely of pure wine or must ; the volatile constituents of which distillate (except water, as provided for in section 15) are derived entirely from the above-named materials, provided that the alcoholic strength of such pure wine brandy be not lower than 25 degrees under proof, and not higher than 22 degrees over proof. "Pure grape brandy" or *grape brandy* means the unrectified distillate resulting from the distillation solely of wine or must with grape-husks, the volatile constituents of which distillate (except water, as provided for in section 15) are derived entirely from the above-named materials, provided that the alcoholic strength of such pure grape brandy be not lower than 25 degrees under proof, and not higher than 12 degrees over-proof. "Dop brandy" means the unrectified distillate resulting from the distillation solely of grape-husks and water, the volatile constituents of which distillate (except water as provided for in section 15) are derived entirely from the above-named materials, provided that the alcoholic strength of such dop brandy be not lower than 25 degrees under proof. "Whisky" means a spirituous liquor derived from grain by fermentation and distillation, the volatile constituents of which (except water, as provided for in section 15) are derived solely from the above material. "Malt whisky" means whisky derived solely from malt. "Blended whisky" means whisky containing not less than 25 per cent. of malt whisky.

* * * * * *

16. No person shall sell under the name of "brandy" or "whisky" any substance except brandy or whisky.

17. The addition to brandy, whisky, or wine spirit of cayenne pepper, tobacco, bluestone, or any substance whose addition is not expressly provided for in this Act shall be deemed a contravention of this Act.

18. The flavouring of brandy other than pure wine brandy or grape brandy, or the addition to any class of brandy, for sweetening purposes, of pure cane-sugar previously made into a syrup (provided that the total quantity of sugar so added does not exceed 1½ ounces per gallon), or the colouring of the same by means of pure caramel or by the material derived from the wood of the cask in which it is stored, shall not be deemed an offence against this Act.

19. No person shall sell, offer, expose, or deliver for sale or authorize, direct, or allow the sale of any Colonial brandy unless the bottle or receptacle containing such brandy is marked, branded, or labelled in such manner as legibly to set forth whether such brandy is brandy, pure wine brandy, pure grape brandy, or dop brandy as defined in section 14.

20. No person shall sell, offer, keep, expose, or deliver for sale as or under the name of Colonial liqueur any article in the manufacture of which any spirituous liquor other than pure wine spirit or brandy may have been used.

21. No person shall manufacture or sell, etc., under the name of Colonial liqueur any article which has not been manufactured either—(*a*) by maceration in pure wine spirit, or brandy of fresh or dried fruit, or peels of aromatic plants, leaves, herbs, roots, or seeds, to which extract has been added subsequently a syrup made of pure cane-sugar or honey; or (*b*) by redistillation of a macerated extract prepared as above, to the resulting distillate of which a syrup made of pure cane-sugar or honey has been added; provided that in no case the alcoholic strength of any liqueur shall be less than 30 per cent. of alcohol by volume.

22. Nothing contained in the preceding section shall be so construed as to prohibit the use of real essence of mint in the manufacture of any liqueur offered or exposed for sale.

GENERAL.

23. No person licensed to sell by retail shall sell, etc., wine, brandy, whisky, or liqueur, unless the bottle or other receptacle wherein such wine, brandy, whisky, or liqueur may be contained is marked, labelled, or branded in distinct letters, with the name and address of both the said retail dealer and, if bottled in the Colony, the

name and address of the bottler, and in the case of imported wine, brandy, whisky, or liqueur, the name and address of the original importer.

24. No person shall manufacture or have in his possession for the purpose of sale, or sell, advertise, offer, keep, expose, or deliver for sale, whether wholesale or retail, or authorize, direct, or allow the sale of any recipe or formula whatever for adulterating any wine, brandy, whisky, or liqueur.

25. Time shall be allowed to enable vendors to dispose of any existing stocks which would otherwise be considered as contravening the provisions of this Act.

26. Gives the necessary powers of entry into any vineyard, storeroom, wine-cellar, bonded or free store, etc.

27. Any purchaser of any article of diet provided for in this Act shall be entitled to obtain from any analyst appointed under this Act, on payment to such analyst of the cost thereof, an analysis of such article by such analyst, and to receive from him a certificate of the result of such analysis, provided that nothing herein contained shall be taken to impose upon any such purchaser the necessity to obtain such analysis and certificate before instituting any complaint or proceeding under this Act if he shall have sufficient evidence otherwise.

28. Any person in charge of any article of diet referred to in section 26 refusing to allow the amount required for analysis to be taken by the person offering to purchase and authorized as hereinbefore provided shall be guilty of an offence.

29. All the provisions of this Act shall apply to wine, brandy, whisky, and liqueur imported into this Colony.

30. No person shall import into this Colony, for purposes of sale, or sell, offer, or expose for sale, within this Colony, under any name, label, brand, or designation provided for in this Act, any article of diet provided for in this Act, which is not of the nature, substance and quality of the article designated by such name, label, brand, or designation, or demanded by any person who may purchase the same.

31. From and after the taking effect of this Act all articles of diet provided for in this Act, imported as merchandise and landed at any port in this Colony may be subject to examination by the Customs or other officers appointed in that behalf, and samples may, when deemed necessary by such officers, be taken and with all convenient speed be examined by an analyst as aforesaid, and if, upon such analysis, the same shall be found to be in contravention of this Act, the same shall be forfeited and destroyed or otherwise disposed of as the treasurer of the Colony may direct.

32. The person acting as analyst in any given case shall furnish a certificate as may be provided for by regulation of the Governor in Council on the recommendation of the Administering Officer and published in the *Government Gazette.*

33. (1) Any person who contravenes any of the provisions of this Act, or of any regulations thereunder, shall be guilty of an offence against this Act, and shall be liable to a penalty of not exceeding £20 for a first offence against any such provision, and in default of payment he shall be liable to imprisonment with or

without hard labour for a term not exceeding three months, unless such fine be sooner paid, and for a second or subsequent offence to a penalty not exceeding £50 and in default of payment to imprisonment for any term not exceeding six months with or without hard labour, and for a third offence to a penalty not exceeding £100 or to imprisonment for any term not exceeding twelve months with or without hard labour, or to both such fine and imprisonment; provided that any violation of the provisions of section 23 shall not be punished by a fine exceeding £5, or in default of payment by imprisonment with or without hard labour for a term exceeding one month.

(2) Every person guilty of an offence against this Act shall, in addition to the penalty imposed, be liable to pay such amount as the Court may award for the costs and expenses incurred in the taking of proceedings against such guilty person, including the expenses incurred by the prosecution in obtaining the analysis.

39. If there be sufficient defence in any prosecution under this Act for the accused to prove that he had no knowledge of the adulteration complained of, and was not a party to it, he shall be discharged from the prosecution.

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REVIEW.

Chemistry for Dental Students. By H. Carlton Smith, Ph.G. (New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1906. Pp. viii and 273. Price 10s. 6d. net.)

As stated in the preface the arrangement of this book follows closely the lecture course in dental chemistry given by the author at the Harvard Dental School. Qualitative and volumetric analysis, dental metallurgy, organic and physiological chemistry, are all dealt with here, in addition to a couple of special sections devoted to digestion and urine. It is hardly necessary to say that with so comprehensive a syllabus each individual subject cannot be adequately discussed within the narrow limits of a treatise of this size. The notes on practical chemistry, given at the commencement of the book, are arranged on the lines of the ordinary separation tables, but it would seem hardly necessary to give no less than eighteen diagrams representing filtration by means of a funnel and beaker.

The subject of dental metallurgy is discussed in thirty pages, whilst only forty-six are allotted to organic chemistry. In the latter we notice a curious misprint, HCl_3 being given as the formula for iodoform.

Although possibly the treatise may prove of value to those attending the author's lectures, it can scarcely be recommended to dental students in this country, where the scope of the examinations in chemistry and metallurgy is of a very different character.

P. A. E. R.

DECEMBER, 1906. Vol. XXXI., No. 369.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, November 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the first time: Mr. Henry William C. Annable, chemist to the Tungsten and Rare Metals Co., Ltd., Queen's Road, Battersea, S.W.; Mr. Bernard Cracroft Aston, chief chemist to the New Zealand Department of Agriculture, Wellington, New Zealand; Mr. James William Brisbane, A.I.C., assistant to Mr. Raymond Ross, F.I.C., Public Analyst's Laboratory, Burnley; Mr. Thomas Cockburn, 148, Woodstock Avenue, Shawlands, Glasgow, chief assistant to Mr. F. W. Harris, F.I.C.; Mr. Archibald Prideaux Davson, A.R.C.Sc. (London), 39, Blandford Square, N.W., assistant to Mr. R. Bodmer, F.I.C.; Mr. David Agnew Griffith, "The Houghlands," Kelsall, Chester, chemist to the River Plate Fresh Meat Co.; and Mr. Vincent Herbert Kirkham, B.Sc. (London), A.I.C., agricultural analyst in the County Technical Laboratories, Chelmsford.

Messrs. W. Dickson and G. W. Monier-Williams were elected members of the Society.

A paper on "The Analyst and the Medical Man" was read by Dr. F. Gowland Hopkins, M.A., M.B., D.Sc., F.R.S.

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THE ANALYST AND THE MEDICAL MAN.

By F. GOWLAND HOPKINS, M.A., M.B., D.Sc., F.R.S.

(*Read at the Meeting, November* 7, 1906.)

In venturing to address you this evening, I am deeply impressed with the sense that the excuse for my intrusion is a small one. Seldom, I imagine, has this learned Society welcomed a visitor less obviously entitled to occupy its time. So fully do I realize this that I must ask you at the outset to listen to a few words of explanation, the personal aspects of which you will kindly excuse.

My appearance here primarily takes origin from the circumstance that I am, at the moment, Examiner in Pharmacology and Therapeutics at the Institute of

Chemistry, happy in occupying the position of such distinguished predecessors as Sir Thomas Stevenson and Dr. Arthur Luff.

Now it is an open secret that the officials of the Institute, and the great majority of professional chemists interested in its examinations, were far from welcoming the imposition of this particular test. It was prescribed by an all-powerful Government Department, and accepted by the Institute as a pill is swallowed by a disbelieving patient. Convinced of the utility of the examination, but believing that some modifications in its details had become desirable, I was allowed to approach the Council of the Institute with some suggestions towards such modifications; and though I did not dare ask for all I should have liked, and though my eloquence was not equal to obtaining quite all that I did ask for, yet it was felt by my good friend the honoured Registrar of the Institute that the views I ventured to lay before the Council might perhaps interest a wider audience, and, at his suggestion, your president and your senior secretary, Mr. Chapman, offered me, on your behalf, the indulgence of a hearing. The flattery of this invitation committed me to the effort of addressing you. But consideration made it evident that anything I might have to say about the examination referred to could not be made to occupy your time with advantage for more than a few minutes, and I felt I must expand this narrow text into something wider, which should more or less naturally arise from it. I came to the conclusion that the only real qualifications I had for coming before you were those possessed by an individual who, having been trained for your own profession, and having acquired some knowledge of its aims and claims, sought later a training in the profession of medicine, and so gained similar knowledge with regard to it. I am, of course, very far from standing alone in this experience; but, unlike many who, with the same doubled training, have practised one or other of these professions with distinction, and, still more unlike those who have contrived to practise both, it has been my fate never at any time to practise either. Even so my case is doubtless not unique, but if anything worth saying can be said about the relations of these two professions, the circumstances, at any rate, give me a claim to speak without prejudice. With no other endowment than what I have indicated, I venture, for lack of other text, to say a few words on the relations, present and future, between the analyst and the medical man.

There is no need, I think, to repeat the indisputable statement that the doctor and the analyst share between them—not forgetting the work of the sanitary engineer—almost the entire burden of the maintenance of public health. It would be but repeating the obvious, moreover, to say that these two professions, having such common aims, should possess full sympathy and mutual understanding. But although we may believe that the sympathy is always present, yet the understanding is not quite always complete, and the lack of it has, now and again—we rejoice to know, not often—led to some friction at points of contact. When such small differences arise it must be admitted that the medical profession has this great advantage over yours—that its claims and its merits are so much more easily understood by the general public. This, should a need for judgment arise, somewhat unfairly strengthens the hands of the one disputant. It must, unfortunately, be recognised that, even now, after all the years during which the Public Analyst has served the community, the layman has a most imperfect knowledge of what his work entails. The services of the engineer are understood, those of the doctor are appreciated, but the skill expended in the analytical laboratory is understood and appreciated but little. This, to you, is again a commonplace; but I believe I am justified in adding to it the really regrettable statement that even the average medical man is not greatly different from the man in the street in this respect. He has at one time known a little chemistry, but he possesses no standard to measure what is required of the skilled analyst. It should be understood that I speak of average cases only.

I am going to take the risk of making a bold generalization here which I must justify afterwards. I believe that, of those professional men who in any sense apply science to practical affairs, the average doctor is among the least scientific, while the professional chemist is among the most scientific. I hope I am not making this statement gratuitously, and I am certainly not urging the contrast to ingratiate myself here, or to cast a slur upon the medical profession. There are many in the latter who are using high scientific knowledge, and applying it in the most difficult of all regions, the treatment of disease; but nowadays the medical man, with a real scientific bent, tends to cleave off from the profession, as a pathologist or physiologist. It is even the fashion, once more, for distinguished medical teachers to urge upon their students that the art rather than the science of their calling is the important thing; and we must all, indeed, recognise that in practical medicine, skilled and wise empiricism, based upon experience, is a much better endowment than ill-digested science. The progress of medicine depends upon science, but its practice does not demand scientific interests, properly so called, on the part of the practitioner. You must not, I think, expect that your work will be understood, or fully appreciated, by the doctor merely because he has passed through the medical curriculum.

But, you will be inclined to say here, the predilections of the doctor as a clinician are of small importance to the analyst, save when the latter as one of the general public is sick and under his hands. The analyst, in the exercise of his own profession, has little to do with the practising doctor; it is with the specialized medical officer of health that his duties bring him more or less into direct contact. This is true, and though it will prove the chief burden of my paper to urge that in the future the professional chemist and the clinician will be in closer contact, the present attitude of the Medical Officer of Health towards the Public Analyst claims first attention.

There is no doubt that in the great majority of cases these two coequal and independent officers of a sanitary authority work together harmoniously, but there have been, in the past, sufficient instances of misunderstanding to make it justifiable to take one's courage in hand and say a word about them, though on such a subject it is difficult, perhaps, to do more than give vent to *obiter dicta*, with the risk of being misunderstood.

The Medical Officer of Health is a man whose education has been wide and various, and who has submitted himself to tests of efficiency which are without doubt rigorous. But although, among many other things, he has been taught more chemistry than the less specialized members of his profession, I venture to think that even he does not—at any rate, at the outset of his career—possess enough chemistry to appraise the high claims of the skilled analyst or to gain the full respect for the latter which comes with knowledge. He does not always recognise, as he should, that the consulting chemist and the analyst belong to a profession in which education is as prolonged, and in which personal ability and skill are as essential, as in his own. I feel that this lack of knowledge on the side of the medical officer is certainly one of the causes leading to misunderstanding. The Public Analyst on his side should not, of course, forget that the Medical Officer of Health is a man of multifarious and responsible duties, whose accomplishments are not to be measured by his knowledge or ignorance of special laboratory problems; he must demand only that these laboratory problems be viewed with a due sense of proportion.

Now we all know that one bone of contention has been the matter of water analyses, which either officer concerned may legally carry out. I believe this is really a somewhat small bone, not really important to the nutrition of either combatant, but it has given rise to some growls. I have in my time taught water analysis to medical students in an official public health course, and if you ask me whether the average man, having finished such a course, and having weathered the examination at the end of it, is equal in practice to examining and reporting upon a series of drinking waters, I must certainly answer, No! The real value of such practical work as he does in class is in enabling him the better to appreciate the significance of analytical data when they come before him. If he subsequently argues to himself, "I have been to the trouble and expense of learning water analysis, and I am stamped by the examiner as efficient: why should my knowledge not add to my guineas?" then, so arguing, he shows himself a man without sense of proportion.

But, remember, there are individual Medical Officers of Health who, owing to special tastes and opportunities, do try subsequently to make themselves efficient in this connection, and it is going much beyond the facts to assert that an intelligent man pre-eminently in touch with practical problems, having carried out routine analyses of a score or so of water samples, is not in a position to claim accuracy for his work so far as it is of this routine kind. You would, if I mistake not, accept the data obtained by your own junior assistants after similar experience.

But the existence of these exceptional cases is beside the mark. To the normal medical officer, even if he be possessed of some skill as a chemist, the chemical laboratory and its pursuits are but an accident. He can enter it only seldom, and if he come into it sample in hand it is usually to find standard solutions which time and accident have made unreliable, and apparatus untested for weeks or months. Nine times out of ten, therefore, the analytical effort is a trying one, and if the medical officer spend the time necessary for accuracy in a laboratory which is not strictly a going concern, the enterprise is to him unprofitable. He should realize how much simpler it all is to the professional analyst in constant practice. No one, I venture to assert, has seen more of these things than I have, and I certainly believe that the medical officer who does not refer all the water analyses which might come his way to his chemical colleague is a person neglecting other, and to him more profitable, opportunities.

But into the controversy about water analyses there came some years ago a certain *tu quoque* argument, and to the disinterested outsider the position was even a little amusing. The examination of water is only partially complete without a bacteriological study; and though it must be admitted that our power to dogmatize about the details of contamination has not yet been aided by bacteriology to the extent that was hoped for, yet it is certain that the water expert must be, on special and limited lines at any rate, a bacteriological expert. But in bacteriological study the medical man for the most part preceded the analyst, and "Hands off!" at first seemed to him as legitimate an expression when uttered on his side of the boundary as when used on the other.

The technique of bacteriology on its cultural and merely diagnostic side is much simpler, more limited, and more empirical than that of analytical chemistry; but the former subject, like the latter, requires something more than a formal knowledge of technique. It is the development of certain special instincts, only given by long contact with the problems, which converts the amateur into the expert. The ordinary training of the medical officer or of the analyst gives in neither case this endowment as regards bacteriology. There is, however, I believe, this fundamental difference between the intrusion of the Public Analyst into bacteriology and that of the medical officer into chemistry: the analyst is first, last, and always a laboratory man, while the medical man is not. The six years of laboratory life which must elapse before the professional chemist is looked upon as fully qualified make the attempt upon new laboratory ventures an easier task for him. This confinement to the laboratory is, on the other hand, the cause of some distinct disadvantages to the analyst. The four walls of his workshop hide him from the gaze of the public; he triumphs over difficulties in secret, and without appreciation. But he should at least reap the advantage of a recognition, from those who know, of the fact that his constant practical experience at the laboratory bench endows him with fundamental instincts for laboratory work in general, which the occasional visitor to the laboratory can only very exceptionally possess.

Some of you have doubtless been practical bacteriologists for as long a period as any pathologist can claim to have been, but the question is now rather as to how far the student who intends to practise chemistry in the future shall spend time and energy on the study of bacteriological technique. It seems to me highly desirable that a certain proportion among chemical students should be encouraged to take an interest in the subject, because of the enormous amount of purely chemical work to be done in connection with it, and because of its growing importance in subjects quite other than medical ones. These are the students for whom Branch E of the Institute's final examination, with its admirable syllabus, is intended.

But as regards the future activity of those who become officers of public sanitary authorities, it is likely that specialization will increase and the bacteriological laboratory will become a unit. I think specialization will here become accentuated, because of the familiar difficulty due to the intrusion of the pathogenic organism. It is, of course, not possible to draw the line sharply between the study of these and the non-pathogenic groups. The *B. coli communis* is an organism which must always concern the water specialist, and yet its case is one which bridges any gulf between the pathogenic and the non-pathogenic. But to follow up the study of the pathogenic organisms fully requires the use of animal experiments, with all their attendant difficulties. The rigour of the license system is likely to increase rather than diminish, and only specialists, working in a comparatively few licensed laboratories, will be able to do useful work. This will lead to the emergence of the bacteriologist as a specialized officer of all sanitary authorities, and since laboratory work is best grouped in accordance with the technique employed in it, rather than in relation to its immediate aims, the specialized laboratory is likely to attract all public bacteriological examinations to its domain. This is my view of the future, and though, for reasons already mentioned, I utterly disagree with Professor Hewlett, for example, when in a presidential address last year he declared that "the present tendency of Public Analysts"—assuming this exists—"to undertake any and every form of bacteriological work is fraught with the greatest danger . . .," yet I believe him to have been right in urging that the bacteriological specialist must emerge in the future. It is highly desirable that the knowledge possessed by any expert should extend beyond the limits of his daily task, and, in fact, that it should be as wide as the shortness of life permits; but it is equally desirable that in the application of expert knowledge to the public service, specialization should go so far as Society and the State can afford.

If in the future the Public Analyst is to be reminded by other specialists that his business is chemistry, the medical officer will be treated on similar terms, and I have firm faith myself that an increase in the analyst's activities, on purely chemical lines, will leave him well content, as I hope in some sort to show later.

There are, it seems, some grounds of complaint against medical officers in other and, to my mind, more serious connections than those yet dealt with. It has occurred that the Medical Officer of Health of a district has acted as though he were the superior of the Public Analyst, in the sense of possessing a right to deal with the reports of the latter, publishing them as though they emanated from a mere departmental officer under his control. Such a distortion of fair conduct, or anything analogous to it, must be and remain rare, and need not be discussed as though it pertained to the normal. It could arise only from the existence of a complete misunderstanding of the situation. I am sure that every reasonable medical officer of health, recognising that the analyst's appointment is a direct one and coequal with his own, would wholly repudiate such a course. In such a case it would be well to see that the facts came to the knowledge of the medical press, in which, I am sure, it would meet with right and effective comment.

Leaving for the moment any further reference to the difficulties which have arisen between doctor and analyst, let me now proceed to the more satisfactory task of indicating future developments which may tend to bring them together.

In pursuit of this side of my subject, I find that I may logically begin by referring to the Institute examination in pharmacology and therapeutics. When this was first established by fiat of the Local Government Board, Sir Thomas Stevenson, the first examiner, very wisely established a standard which has since kept the range of the examination within certain narrow limits. The student has been expected to recognise by their naked-eye characteristics the various drugs of the British Pharmacopœia, and to know roughly the practical uses of the more important drugs, and the doses, medicinal and fatal, of such drugs as are presumably toxic.

Such limitations were very necessary on the first establishment of the subject, and the present view of the Council, which, with certain modifications, should, I think, be respected by all wise examiners, is in favour of the careful avoidance of any demand for knowledge within the proper province of the medical student.

At the moment, however, things have arrived at this pass: Either as a result of the growth of coaching, or because of present organized facilities for handling pharmaceutical preparations, not a single candidate now fails to be well versed in the recognition of drugs. It is most rare to find a single individual who cannot, with the aid of various "tips" and *memoria technica*, recognise with unfailing accuracy every typical drug which he may be shown. His knowledge of doses is equally accurate, for he comes provided with a short list which has been got by heart the night before, and can be again consulted at the eleventh hour. Indeed, on the somewhat rare occasions when it becomes necessary to refer a candidate, it is almost always, in my experience, because of weakness in the associated subject of microscopy, in the practical technique of which I have been surprised to find a chemical candidate inferior to the medical student of similar standing.

Now, as examiner, I certainly did not wish for more opportunities of referring candidates, nor did I desire to increase the amount of work the student has to do. In asking for a somewhat more extended syllabus, I had several considerations in view which I should like to submit to you.

In the first place, I found the candidates come up for this examination with feelings of considerable discomfort. So far as the matter of previous papers set on the lines defined above can guide them, they succeed easily in making themselves letter perfect; but they feel that between the boards of a text-book of pharmacology and therapeutics there are many things beyond these, and though they understand they are not to be treated as medical students. they yet feel uncertain as to what exactly may be sprung upon them, to their no small distress. In circumstances such as these a syllabus which seems to extend the subject may really limit the amount of reading to be done.

Next, it appeared to me that if a given subject is studied under compulsion, those parts of it which stimulate interest and are of educational value should certainly not be eliminated, and it seems to me that "spotting" drugs and learning doses by heart possess little of either of these qualities.

Again, there seemed to be aspects of pharmacology which really concern the professional chemist as much, if not more than, those hitherto emphasized. I have found, for instance, that candidates who can without hesitation distinguish between caraway and dill, or rattle off a list of doses, did not know that a man taking chloral hydrate excretes a product which, by reducing Fehling's solution, has led to confusion with glycosuria. Yet he might quite conceivably be face to face with the results of this phenomenon in his laboratory, and such matters, as well as many analogous ones, seemed to me to concern him even more than a knowledge of poisonous doses. Moreover, if the fatal dose of a poisonous drug is supposed to be known, there should certainly be the added knowledge of the influence of age, idiosyncrasy, and habituation as modifying such doses.

Questions as to whether a poisonous drug is rapidly eliminated, or whether it is accumulated in the body, and whether, when it has to be looked for, it will be found unaltered or changed by the extraordinarily interesting processes to which the body may submit it, are of import to the professional chemist; for, though it is to be hoped that investigations concerning criminal toxicology may largely remain in the hands of specialists, yet the help of the professional chemist in ordinary practice may often be sought in non-criminal cases. His help may, indeed, become more important, and at the same time his task somewhat more difficult, with the growing use of complex organic drugs.

It further seemed to me, though I was not allowed by the Council to insert such matters in the syllabus, that a chemical student who is compelled to learn pharmacology at all might well have his attention called to the interesting relationships which exist between the chemical constitution of active substances and their effect upon the body. This knowledge, and the synthetic work based upon it, has certainly proved highly profitable to scientific chemists in Germany.

Such knowledge as is asked for in the present syllabus can be got from almost any reasonably complete text-book by selecting a few special sections. It is not its bulk, but its suggestiveness, that gives it importance.

Note that it has not hitherto been my purpose to discuss the wisdom of the actual institution of a professional examination in pharmacology and therapeutics; but, taking it as an accomplished fact, I have wished to point out that, without taxing the student's time overmuch, and without treating him in any sense as a medical student, he may with great advantage be asked to know something more than the dry bones of the subject.

But I believe further that, just as the establishment of Branch F (biological chemistry) came at an opportune moment, and will help to provide experts whose services will be of great value to the State in the immediate future, so the existence of a section in Branch E capable of giving some slight medical bias to the minds of even a few students will ultimately prove of no small service, both to themselves and to the medical profession. This is my firm faith, and this is why I have wished to see the examination deal with something more than the dead and unstimulating aspects of the subject. I will try to explain why I hold this faith in the value of a medical bias just now.

As many of you will have realized, there has been enormously accentuated progress in physiological chemistry during quite recent years. The new knowledge being gained is living stuff and of real practical importance. Now, progress in physiology rapidly reacts upon pathology, and pathology upon methods of diagnosis. Pathology is striking out some chemical paths of its own, but it has not yet felt the effect of the unloading of chemical facts which physiology is preparing for it. Sooner or later there will be activity on lines unknown to the physician at present.

If we consider the question of urine analysis alone, it is easy to prognosticate that there will be added to the study of the few constituents which are now troubled about that of a great number of excretives, appearing in small amount, but of great importance as measures of departures from the normal metabolism. The practical urine analyst of the future will have to cover an extensive ground. Even now, with the physician only vaguely aware that the pathological chemistry of the past, which was too ill developed a subject to be of much use to him, is giving way to something more real—even now there is some awakening to the importance of the laboratory. On the Continent, at any rate, and especially in France, elaborate and very numerous analytical studies are continuously made by professional analysts, from which characteristic curves are constructed, these being supposed to give important indications as to departures from normal health. Though many physicians in France attach much importance to these curves and attribute to them even a prognostic value, I believe myself that much of this work is only pseudo-scientific; it is a little premature and undiscriminating, but it is, at any rate, exceedingly profitable to the analyst, the patient—if not the physician—cheerfully paying satisfactory fees.

In this country the medical profession is, at any rate, beginning to think more seriously of the professional laboratory as an aid to its work. This is evidenced by the astonishing success of certain medical investigation associations run upon business lines. Only part of their work is chemical, however, and as the complexity of the chemical problems grows, the special difficulties of chemical technique will send the work more and more into the hands of individual experts.

The average medical man, while not yet aware of all that chemistry can do for him, is also, for reasons already indicated, not yet clear as to whom he should turn to for help in those chemical problems which he has, even at the present time, in mind. I have read recent elaborate medical monographs, involving extraordinarily futile chemical investigations, in which the author, himself innocent of chemistry, acknowledges his obligations to the pharmacy which is adjacent to him. Now, it is no reflection upon an honourable calling to suggest that in such a case the doctor ought to have gone elsewhere. One can see that in these conjoint researches it was the good nature of the pharmacist, and no worse motive, which made him consent to the unprofitable partnership. But I believe it is not going too far to say that the rise of chemical pathology to its full importance will call almost for a new profession.

I wish very much to make a point here which, if it seems too remote at present from your practical interests, may be considered as in parentheses. The care of the body in sickness, with all the delicacies of human relationship which it involves, must remain always an entire and carefully guarded prerogative of the physician; but the innate respect of the public, and even of the non-medical scientific public, for the physician's calling has led to a somewhat illogical attitude, and has tended to make sacrosanct not only the calling of the physician, but the scientific material which he deals with. There has been, as it were, an averting of the gaze whenever a region of knowledge is stamped as "medical." Now, I am certain that the progress of scientific medicine demands a change here. While a large part of future scientific medical studies must always be carried out by men who, though medically qualified, have preferred the laboratory to practice, and whose special qualification, therefore, is that they have had personal touch with the problems offered by disease, yet in a middle region these must be joined in their work by men whose primary qualifications are non-medical—men who, saved from the long years of clinical study, are able to bring well-grounded laboratory knowledge and (I may add) a sufficient knowledge of the literature of pathology, which is open to all, to join their medically qualified *confrères* in attacking the huge problems which await solution.

Many of these must be organic chemists, and, to make a distinction which I will urge again later, many of them must be also analysts. If it be felt that these are matters which concern those who aim at pure science, and not the professional chemist, it is yet certain that the amount of valuable analytical work to be done professionally and outside the research laboratory will greatly increase as a result of the tendencies indicated.

My own calling compels me to know well the present position of physiological and pathological chemistry, and I may claim to have had first-hand acquaintance with the large amount of paying work which is even now being done for the medical profession, though not by individual professional analysts. And though I may not stop to prove my point further, I yet reiterate my opinion that there will be in the near future an efficient source of income for many of the students in your laboratories if they are prepared to work on these lines.

While upon the business of prophecy, I am tempted to put another series of prognostications before you, the credibility of which is at the present time, perhaps, more obvious to the physiological chemist than to anybody else. I pass from pathology to an aspect of dietetics. This is a subject in which the medical man is the recognised authority, charged with instruction of the public, but for a scientific knowledge of which he depends largely on the chemical physiologist and the analyst.

Putting on one side the aspect of affairs which especially concerns this Society—the maintenance of purity and freedom from adulteration—and leaving out questions such as digestibility and the like, the chief practical points which have hitherto been considered in relation to the daily rations of mankind are the total energy value requisite for maintenance, the optimum ratio of fats and carbohydrates, and the optimum supply of protein. Now, these questions have recently received fresh attention, and experimental work has been done lately yielding, as you know, somewhat startling results, tending at first sight to modify our views concerning maximal, minimal, and optimum dietaries. But I am not going to discuss the work of Atwater or Chittenden, proposing rather to put before you very briefly facts of another sort, less known and seemingly academic. I believe, however, that my theme, which is that of the influence of minimal qualitative variations in dietaries, will one day become recognised as of great practical importance.

Physiological chemistry, chiefly owing to the work of Emil Fischer, has recently gained the knowledge that individual proteins, and among them those which contribute to human dietaries, may each bear a special chemical stamp; that a given protein may differ so widely from another protein as to have, quite possibly, a different nutritive value. I will illustrate this, first of all, by a somewhat extreme case. A protein, zein, forming no inconsiderable proportion of the total nitrogenous constituents of maize, is entirely deficient in at least one characteristic molecular grouping. It yields on digestion no tryptophane, the product which represents the indol group present in the molecule of most typical proteins.

In mentioning tryptophane, I cannot deny myself a moment's harmless gibe at your expense. The well-known colour reaction which you have used for so many years as a test for formaldehyde in milk is really a reaction due to this indol group of the casein. Now, as it was a similar colour reaction which led some of us at Cambridge to separate the tryptophane of protein for the first time, I have felt that some of you, being authorities on food-stuffs, ought with proper enterprise to have anticipated us in this not unimportant discovery.

Recently we have fed animals with this indol-free maize protein in such a way that it formed the only supply of protein, though associated with abundant fat and carbohydrate and suitable salts. The diet wholly failed to maintain tissue growth in young animals, which, however, grew at once when their zein was replaced by pure casein. When tryptophane was added to the zein diet, there was still inability to maintain tissue growth, doubtless because the zein has other deficiencies as a protein. But now an interesting fact came to light. The animals which received the missing indol derivative in addition to the zein did not grow, in fact, continued to lose weight daily, and were afterwards in much better health than, and long outlived, those which had the zein alone. These experiments seem to show two important facts: First, that in an extreme case a particular protein may wholly fail to support life, just as is the case with gelatin; and next, that a group in the protein molecule may serve some purpose in the body other than that of forming tissue or supplying energy. The usual discussions about food-stuffs attribute to them these two functions only—repair of the tissues and energy supply. But the body has other and more subtle needs equally urgent. Here, there, or elsewhere in the organs must appear special, indispensable, active substances which the tissues can only make from special precursors in the diet.

The indol grouping in the protein molecule serves some such special purpose, quite distinct from its necessary function in tissue repair. This matter of qualitative differences in proteins may be of no small significance in dietaries. It may account for what I believe is proved by experience—that rice may serve the races which rely upon it as an almost exclusive source of protein, while wheat is only suitable for races that take a much more varied dietary. It may explain many variations in nutritive values which at present we feel and recognise only vaguely. In the future the analyst will be asked to do more than determine the total protein of a food-stuff; he must essay the more difficult task of a discriminative analysis.

But, further, no animal can live upon a mixture of pure protein, fat, and carbohydrate, and even when the necessary inorganic material is carefully supplied the animal still cannot flourish. The animal body is adjusted to live either upon plant tissues or the tissues of other animals, and these contain countless substances other than the proteins, carbohydrates, and fats.

Physiological evolution, I believe, has made some of these well-nigh as essential as are the basal constituents of diet. Lecithin, for instance, has been repeatedly shown to have a marked influence upon nutrition, and this just happens to be something already familiar, and a substance that happens to have been tried. The field is almost unexplored; only is t certain that there are many minor factors in all diets of which the body takes account.

In diseases such as rickets, and particularly in scurvy, we have had for long years knowledge of a dietetic factor; but though we know how to benefit these conditions empirically, the real errors in the diet are to this day quite obscure. They are, however, certainly of the kind which comprises these minimal qualitative factors that I am considering.

Scurvy and rickets are conditions so severe that they force themselves upon our attention; but many other nutritive errors affect the health of individuals to a degree most important to themselves, and some of them depend upon unsuspected dietetic factors.

I can do no more than hint at these matters, but I can assert that later developments of the science of dietetics will deal with factors highly complex and at present unknown.

But am I at present justified in troubling you, as practical men, with such matters—you who are interested in professional chemistry, and not in what is still more or less academic physiology?

I have been led to do so from two considerations. First, it is abundantly clear that the foundation of future progress in chemical pathology and dietetics on the lines I have been indicating calls for large efforts in purely analytical chemistry—efforts which have been too long delayed. And the delay has arisen from a circumstance of no small interest and importance.

The scientific chemist—unlike his predecessors, the pioneers of sixty or seventy years ago—has long ceased to be much interested in the animal or the plant. Further, the triumph of synthetic work in advancing theory has led the pure chemist away from the especial difficulties of analytical work. His extraordinary developed technique concerns itself only secondarily and imperfectly with analytical studies of the kind still necessary in physiological problems. I mean the endeavours to identify and separate unknown substances, with unknown properties, present in complex mixtures. Only now and again has he made special efforts in this direction, such as that with which Fischer started his work upon proteins. Such work really requires special instincts, and the pure chemist has largely lost them. He is but a poor analyst, as the physiological explorer finds on turning to him for help. I feel that this help, so far as the immediate future is concerned, will have to come from the pupils primarily trained in your own laboratories, where the analytical instinct is developed. Some of your students, it is to be hoped, will have their attention turned in this direction, and to at least a few there may ultimately come opportunities for research; for research, in all callings, even that of the academic teacher, is only to be snatched from leisure. There are the beginnings just now of a renewed interest in biology on the part of all chemists. May the analyst feel this too. It is not only the manufacturer and the sanitary authority that require his help.

In the second place, I am not afraid to assert that progress in dietetics, no less than in chemical pathology, is about to react largely on professional chemical practice. Fresh problems and new ideas will unfailingly extend the field of professional operations.

All progress of the kind I have been hinting at cannot fail to be of the greatest importance to the doctor; and if I may seem to have maligned him in previous paragraphs, I know well how ready and able he is to make use of all knowledge that he believes to yield advantage to his patients.

I see abundant reasons for believing that in the near future events will march to the consummation of mutual appreciation and helpfulness, and to the disappearance of all misunderstanding, in the relations between analyst and medical man.

DISCUSSION.

At the invitation of the President, the discussion was opened by

Dr. BUCHANAN, of the Local Government Board, who endorsed the remarks of Dr. Hopkins as to the advantages which might be derived from a closer association of the analyst with the medical consultant and the medical practitioner. Dr. Hopkins also saw, as probably they all did, many opportunities for a greater use of the chemist in matters relating to the public health. He might make a few observations on this matter, although it was not, perhaps, the part of the paper on which Dr. Hopkins would wish to lay greatest stress. Some of the considerations to which reference had been made—the lack of appreciation of the work of Public Analysts under present circumstances, the isolation of the analyst as a "laboratory man," and especially the limited use that was at present made of chemistry in relation to public health questions—were, it should be remembered, arguments that were used in support of the suggestion that was very frequently made in the larger municipalities, and was coming more and more to the front, for the appointment of whole-time municipal chemists and analysts. Those authorities said that they would like to put their chemical service on the same footing as other expert services, and to have a chemical officer on the spot, who would not only do formal work under the Sale of Food and Drugs Acts, but would undertake research work and other chemical work for the municipality, and would be able to advise and confer with the executive officers as to the collection of samples, interpretation of analytical results, and so forth. It must be recognised that in some aspects there were advantages in this solution of the question. He was far from saying that it was the right one, even for the larger cities and boroughs, but he thought that one of the legitimate reasons for this proposal was that under such an arrangement better use might be made of chemistry in relation to public health. Assuming, however, that the Public Analyst continued, as now, to occupy a position which in many ways stood apart from that of other municipal expert officers, the question arose, How could his work best be utilized and developed in the interests of the public health? The obvious answer seemed to be, By full and cordial co-operation between the analyst and the medical officer of health who was on the spot. Dr. Hopkins had said that medical men of scientific bent were wont to specialize—in physiology, pathology, and so forth. There was another thing in which they specialized, namely, public health administration, and where the analyst had the opportunity of working with a trained officer of that kind it was of the utmost benefit to both that their mutual relations should be as cordial as possible, and that they should help one another. If he might find just a little fault with this most admirable paper, it was that it seemed to him in the first part to go a little out of the way, not to emphasize the necessity for such co-operation, but to draw attention to small and admittedly trivial points of divergence and difference between Medical Officers of Health and Public Analysts. With regard, for instance, to water analyses, he ventured to say (speaking for the moment of those not holding combined appointments) that no Medical Officer of Health undertook water analysis willingly. He would much rather leave it to the Public Analyst. But in many cases the position was forced upon him by the local authority, and this was not a grievance against the Medical Officer of Health, but against the local authority. Occasional differences and even stupidities did, of course, occur, as he himself knew, from both sides. For example, there was the Medical Officer of Health who said to himself—say in reference to samples of canned meat—"I will give the Public Analyst no information at all," and straightway stripped the tins of all their labels and sent them to the Public Analyst under mere numbers; whereas, of course, the whole point was to get information and get the views of the Public Analyst, and to let him have every possible help and information. On the other hand, there was the analyst who said, "I am not going to supply the medical officer with any information, or to tell him what my results are for individual milk samples, and, in fact, am not going to send him any reports at all, because it is not laid down in the Sale of Food and Drugs Acts." Just now, when the assistance which chemistry was able to afford in regard to public health was becoming more and more apparent, and when the medical officer and the Public Analyst were coming more into contact, these points of friction occasionally arose, but he thought they were much more trivial than to the people immediately concerned they sometimes appeared. He thought that Dr. Hopkins quite realized that they were exceptions, and, although they received a good deal of attention momentarily, he did hope that too much stress would not be laid upon them. He believed that Medical Officers of Health and Public Analysts, as a whole, were fully alive to the necessity for "give and take," and that they would not allow friction to arise which would hinder the work which they did for their mutual benefit and that of the public. He was very glad to have had the opportunity of hearing Dr. Hopkins' observations on the probable development of the work of the analytical chemist in the investigation of proteid food constituents. The paper had in many ways given them a great deal to think about.

Professor R. T. HEWLETT (King's College, London) said that on the whole he was in cordial agreement with Dr. Hopkins' remarks, and any criticism of them that he would have been disposed to make had been admirably put by Dr. Buchanan. With regard to his recent presidential address, which had been referred to, he did not think that Dr. Hopkins' quotation was altogether fair, because the remarks quoted were very much qualified by what followed them. The view which he took was that the analyst was not in a position to deal with pathological problems. It seemed to him that many chemists had no biological training at all, and most had certainly no training in pathology; and when problems of disease had to be dealt with, as was the case in many of the examinations that had to be made to-day, he thought it distinctly dangerous to the public health that those examinations should be undertaken by anyone who could not view pathological problems in the light afforded by a medical training. He believed that the position had been partly thrust upon the analyst. One instance which he had in mind was that of a very important city which insisted that its recently appointed Public Analyst should undertake examinations for diphtheria, typhoid, tubercle, etc. To his mind, that was wrong. It seemed to him that in an ideal sanitary administration each department should have its analyst—*i.e.*, that there should be both a chemical analyst and a bacteriological analyst, and for the latter a medical training would probably be desirable. The work of the analysts, with that of the sanitary engineering staff, would be co-ordinated by the Medical Officer of Health. With regard to the examinations of the Institute of Chemistry, there was just one addition that he should like to see made to the excellent syllabus of the examination in biological chemistry. As matters stood at present, candidates could enter for the examination without previous biological training. Such training was essential to the proper taking up of work of that kind, and he thought that the Institute would be well advised to insist upon it.

Mr. HEHNER said that analysts owed gratitude to Dr. Hopkins for having pointed out directions for research and means by which their professional knowledge could be more useful to physiologists and their medical colleagues. It was singular that, as far as he knew, not a single University chair had been established for the teaching of analytical chemistry in this country; and as one who had always insisted that the analytical chemist, in order to take his proper place amongst chemists, must be a highly-cultured scientific man, he deplored the fact that at the Universities no specific notice had been taken of the claims of analytical chemistry as a science. He thought that the cases referred to by Dr. Hewlett, in which analysts were forced by ignorant local authorities to undertake pathological work, must be very rare; and that if the medical profession, which was generally well represented on local authorities, were so minded, they could easily prevent such undesirable appointments. Analysts did not want to do work outside their function, but if it were forced on them they would in some cases submit, just as Medical Officers of Health had sometimes, perforce, to undertake chemical analytical work against their inclination. The time when every local authority of sufficient magnitude would have a chemical officer as well as a medical officer was beyond doubt approaching. Although its advent might damage some members of the analytical profession as at present constituted, the damage would be insignificant compared with the resulting public benefit. The analyst had a claim to be recognised as an independent officer of public authorities, working, whenever the subject demanded, in conjunction with the medical department, but dealing in a great number of cases with chemical problems quite foreign to medical or public health matters. He (Mr. Hehner) had always been most anxious that the analytical profession should maintain as many points of contact as possible with their medical friends and colleagues, but that under no circumstances should a chemist be considered as an officer working under the Medical Officer of Health, and that in an age which owed at least as much to chemistry as to medicine the chemist was entitled to have the recognition of his independent position. The sooner combined medical and analytical practices were things of the past the better. Only on one point was he inclined to differ from Dr. Hopkins. While it was quite true that the ignorant public had still a great deal to learn about the useful services which the analytical profession could render, and that many people, instead of going to the chemist direct, still referred questions to the medical man, on which he at best could have but second-hand information, the fact, nevertheless, remained that, in the twenty years during which the profession of chemistry had existed in this country, enormous strides had been made. The position of the chemist at the present time, compared with that before the foundation of the Institute of Chemistry, was incomparably better. The extent to which practising chemists had multiplied, and the fact that they were, as a whole, at least as prosperous as the average medical practitioner, afforded proof of the progress made.

Professor BOSTOCK HILL said that he happened to be a county Medical Officer of Health and a Professor of Hygiene, but he had also been a Public Analyst for over thirty years, and therefore felt that he might perhaps look at this matter from points of view that were not open to some others of the members of the Society. He should like to say how much he appreciated practically all that Dr. Hopkins had said. He thought that if any jealousy existed between the medical man and the analyst it was largely due to two factors: Firstly, the special work of the Public Analyst was largely an inheritance of work that had originally been done—what little of it was done—by members of the medical profession. One could not forget that in the early days much of the work that formed the basis of the modern Public Analyst's work was done by medical men, of whom Hassall, Letheby, and many others, might be mentioned as notable examples. Another factor was, to use Mr. Hehner's words, the profound ignorance, not only of the public, but also of medical men, as to the duties of the analyst, and sometimes, he believed, ignorance on the part of the analyst as to the functions of the medical man. The public had been accustomed to look upon the medical man as almost the symbol of scientific work, and on that account expected him to do things quite outside his province. The Medical Officer of Health, who could be taken as the type of medical man chiefly concerned in this discussion, might be said to be in a state of transition—to be undergoing evolution. When Medical Officers of Health were first appointed their work had practically to be developed by themselves. Now their work was on a fairly firm basis, and was developing on many lines. The same might be said in the case of the Public Analyst. He (the speaker) did not think that there should be the least jealousy between the two. If any had arisen, it was largely through their want of appreciation of each other's work. If the Medical Officer of Health sometimes assumed an attitude of apparent superiority towards the Public Analyst, it was not altogether surprising, when it was remembered that at the present day, whether rightly or not, the Public Analyst was practically a worker in the department of the Medical Officer of Health as far as public health work was concerned. Therefore in deciding, for instance, what samples should be taken, it was only natural under present circumstances that, in the case of a large authority, the Medical Officer of Health should have control, without, of course, in any way interfering as regards the results or conclusions of the chemical work. As one in touch with medical teaching, he felt convinced that, while the Public Analyst might do certain bacteriological work with advantage to himself and to the public, yet any entry of his into work of a pathological kind would be resented by the medical profession, and would not lead to any good. Attempts were certainly made in this direction; and he knew at the present time a Public Analyst who, willy-nilly, had to do all kinds of pathological work that might be sent to him from a very wide area. It was not easy for the analyst to refuse, and such cases could probably only be met by a strong professional feeling that work appertaining to those sciences comprised in the medical man's work should be kept, at all events, in the medical branch of the profession. He had had some experience of examinations in pharmacology, etc., and he thought that, in the case of the Institute examination in its present form, a man might, given a fair amount of daily time, learn all that was required for the therapeutical part in three or four weeks. That a man should be labelled as having a knowledge of therapeutics after so short a period of necessary work was, he thought, not at all of value to the profession of chemistry.

Mr. F. J. **Lloyd** said that he ventured to think that it was quite as difficult for the Medical Officer of Health as for the Public Analyst to undertake pathological bacteriological work, and his own view was that in the future such work ought not to be put upon the shoulders of either, but ought all to go to specialists. There was, however, a large quantity of non-pathological bacteriological work that could be done by either the one or the other; and he agreed with Dr. Hopkins that the man who was always in the laboratory was better able to undertake that work than one who must necessarily leave his laboratory very frequently, and perhaps for long periods. He thought, therefore, that in the education of the analyst non-pathological bacteriology should be given a prominent place. Dr. Hopkins had made some interesting and valuable suggestions as to the need for investigation in certain directions. He (Mr. Lloyd) would like to mention one instance showing how difficult such investigation was. There was abundant evidence that the disease known as "Barlow's disease" resulted from the feeding of children upon boiled milk, and entirely disappeared on reverting to the consumption of fresh milk. Now, the analytical differences between boiled and unboiled milk were very slight indeed, and their investigation would probably require some years of very careful work at great pecuniary expense. Could any analyst dependent for his income on his analytical practice be expected to give the necessary time and money to such a research? He thought not, and therefore agreed with Mr. Hehner that it could not be hoped that such recondite subjects would be fully investigated until the Universities were able to appoint professors charged with the duty of investigating, and of training others to investigate, analytical problems which were far beyond the ordinary routine work of the profession.

Dr. **Voelcker** said that he was hardly in agreement with Dr. Buchanan in thinking that the points touched upon in the first part of the paper were perhaps somewhat trivial. These were the points which really came into prominence when the relations between the medical man and the analyst were discussed, and for a considerable time they had been a cause of agitation. The remedy for them, and for the harm caused by the continual search after cheapness, must, as had been pointed out, be looked for in the better education of the public. In this connection it would be wrong not to take the opportunity of acknowledging the work of the Institute of Chemistry in emphasizing the need for keeping separate these two classes of work, and for urging that the claims of both should be fairly represented. The Institute, urged on by its energetic Registrar (whose presence they were glad to have at that meeting), had been steadily insisting on the importance of this matter, and had made repeated representations on it to the Local Government Board. The tendency was for a man to rise to what was expected of him, and to try as best he could to do what the public or the local authority thought he ought to be able to do. The result, however, was often unsatisfactory. It might be pointed out that the Institute's examination in branch "E" was framed in accordance with the desire of the Local Government Board. It would, however, be wrong for anybody to take advantage of having passed that examination and say that he was therefore qualified to take up bacteriological and similar work. The chemist could not afford to ignore the help that was to be obtained from bacteriological study, but it was quite another thing to pose as an authority on the subject; at the same time, it was true that the chemist, from his constant work in the laboratory, had better opportunities of learning bacteriological methods and applying them than the medical man had of picking up the details of chemical analysis.

Mr. FAIRLEY remarked that, while the analytical profession was undoubtedly indebted to the work of medical men in the past, it was equally true that much help had been given to medical science by chemists. Pasteur was a conspicuous instance, and there were many others. With regard to the "clinical research associations" which had been referred to, he thought that the very success of these institutions, where the reports of the work done by various professional men were sent out merely signed by a secretary, must tend to deter young chemists from giving attention to problems of pathological chemistry.

Mr. C. T. KINGZETT said that, although neither a Public Analyst nor a Medical Officer of Health, he had devoted a good deal of attention to public health questions; and he felt very strongly that all chemical matters should be referred to the chemical officer, and that the medical officer should no more encroach upon the domain of the chemist than the latter should encroach upon the domain of the medical officer. He thought that a good deal of confusion arose in this matter from a misuse of terms. It was most regrettable that the Public Analyst should be so called; he ought to be styled the Chemical Officer. The subject of chemistry was so wide, and covered such a large range of knowledge, that to call a man a Public Analyst did not adequately describe the position which he ought to occupy in relation to public health. Dr. Hopkins seemed to consider that the acquaintance of medical students with bacteriological work must be more intimate and thorough than that of the chemist. He (Mr. Kingzett) did not think that that was at all the case. He thought that the chemist who followed bacteriological work was much more likely to have an adequate knowledge of it than the ordinary medical student or practitioner. The difference between the effects produced by so-called pathogenic and by non-pathogenic organisms was all a matter of chemistry, and the chemical knowledge which the medical man obtained from his ordinary course of instruction was absolutely inadequate to enable him to draw any sound conclusions in this branch of work. The work of the medical officer should, if possible, be restricted to matters pertaining to the outbreak and prevention of the spread of disease; and if there must be an officer in a position of control, he should be an "Officer of Health"—not necessarily medical, but at least an expert capable of assimilating the knowledge to be derived from both sources, and of presenting it in a proper and digested form to the municipal authority.

The PRESIDENT (Mr. Bevan) said that he was very glad that Dr. Hopkins had laid so little stress upon the so-called antagonism between the Medical Officer of Health and the Public Analyst, which he thought was very much overrated. Certainly it had never occurred in his own work, and he thought that a good deal of it was due to petty jealousies that ought not to exist. Although not disposed to go so far as Dr. Hewlett, he agreed with Dr. Hewlett in thinking that Public Analysts had no right to trench upon the province of bacteriology; but he did consider that they ought all to be acquainted with what might be called the more rudimentary methods of bacteriological examination. He was glad also that so little had been said about dual appointments. He was free to confess, and did so with a full sense of responsibility, that the ideal Public Analyst was a medical man with a great knowledge of chemistry. But men like Sir Thomas Stevenson were very rare, and in these days of specialization were likely to be rarer still, so that it was highly important that the two offices should now be kept distinct.

Dr. Hopkins, in reply, said that he had felt considerable compunction in reading this paper before the Society, even after it had been written. He had a strong feeling that, while the first half was made up of platitudes and indiscretions, the second consisted of an academic lecture. Still, as regards the points first touched upon, it was, perhaps, difficult to utter other than platitudes, while he was bound to say that he did not wholly regret the second part, for, if anything gave him pleasure, it was to talk academically to practical men. He might have made too much of the points of difference between analysts and medical men, but he had been tempted to try and make a contrast between the present state of things and the Utopia which might exist. Although he still did not agree with Dr. Hewlett, he owed him some apology for having concluded his quotation marks a little too soon. Dr. Hewlett had gone on to say that bacteriology in the hands of the Public Analyst would be a very dangerous thing "unless the chemist made himself a biologist or employed in the laboratory those who had had biological experience." That addition to the quotation perhaps made all the difference, but he still thought that there was not the least danger in trusting the Public Analyst with a pathogenic organism, for the reason that he believed a laboratory man to be a safe person. It was quite right, as Dr. Hill had said, to recognise what the medical profession had done in the past, but the time had long gone by when the medical man was the only scientific man available. The question of the Institute examination was, of course, one which he had very much at heart, and, with regard to the views thereon of Dr. Voelcker and Dr. Hill, he should rather like to know whether it was before or after his suggested alterations that they did not think much of it! With regard to the difficulties of entering into research to which Mr. Lloyd had referred, he had felt that the original work that was done in strictly analytical laboratories was, perhaps, in too few hands. The provision of chairs of analytical chemistry at the Universities was, he believed, at any rate as far as Cambridge was concerned, merely a question of money. But research in any event had to be stimulated, and he thought it important that students of analytical chemistry should be encouraged, as part of their advanced work, to make original investigations bearing upon their special branch of applied science. One would wish that the Institute of Chemistry might ultimately see its way to making some sort of award for the encouragement of research.

The PRESIDENT remarked that, under the research scheme initiated some time ago by the Society's Council, there had been sent to various Universities a great many problems, the investigation of which had been undertaken, under the guidance of their professors, by senior students. He then moved a vote of thanks to Dr. Hopkins, which was heartily carried.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Sugar contained in Milk. J. Sebelien. (*Festschrift zum 65-jähr. Geburtstag von Olaf Hammarsten*, No. 17; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 337.)—The author shows that a pentose, probably arabinose, is present in milk to the extent of about 20 to 40 mgms. per 100 c.c. of milk. Other unknown carbohydrates must also be present to explain the differences always found between the gravimetric and polarimetric determinations made on the milk serum free from proteid, and the different values found when the quantities of Fehling solution used are varied. A. G. L.

A Rapid Method of Removing Alcohol from Ether. P. Guigues. (*Journ. Pharm. Chim.*, 1906, xxiv., 204.)—When a mixture of alcohol and ether is distilled with a resin such as colophony, the alcohol is retained by the pasty residue left in the flask. Thus, on distilling a litre of ether containing about 1 per cent. of alcohol with about 50 grams of colophony, a distillate is obtained free from alcohol.

C. A. M.

Formic Acid as Preservative. G. Lebbin. (*Chem. Ztg.*, 1906, xxx., 1009.)—From the author's experiments it appears that the addition of 0·15 per cent. of anhydrous formic acid acts as a perfect preservative for all manner of food-stuffs, more especially fruit extracts and syrups. In most cases 0·10 per cent. even is quite sufficient. As regards toxic properties, reasoning from experiments on rabbits, the author finds that it would take a dose of 3·5 grams of formic acid to produce harmful results on an adult human being. From actual experiments on himself and his assistants, he finds that a daily dose of 0·5 gram formic acid, taken regularly in the form of lemonade for two to four weeks, was without the slightest effect. He believes that it is perfectly safe to assume that formic acid is not more than twice as poisonous as acetic acid. A. G. L.

The Action and Efficacy of Preservatives. A. Behre and A. Segin. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 461-467.)—Tabulated results of experiments with various preservatives are given. Definite quantities of the preservatives were added to unsterilized meat-juice diluted with water, and observations made as to the change in colour of the solutions as well as to the time taken before an odour of decomposition was noticeable. The results show that formaldehyde has by far the most powerful preserving action, that benzoic acid is a better preservative than salicylic and boric acids, and that sodium benzoate is about equal to boric acid, whilst sodium thiosulphate and sulphite are very feeble preservatives. W. P. S.

Gravimetric Determination of Potassium Nitrate in Meat. C. Paal and G. Mehrtens. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 410-416.)—The method described is an application of Busch's process for the determination of nitric acid by means of "nitron" (see ANALYST, 1905, p. 256). Fifty grams of the finely-minced meat are agitated with warm water for about two hours, the mixture being then brought to boiling and the solution filtered. The residue is boiled out with several successive small quantities of water until the extracts no longer give a reaction with diphenylamine, and the united extracts are then diluted to a volume of 500 c.c. Two hundred c.c. of the solution are now evaporated to a volume of about 50 c.c. When quite cold, 3 drops of ammonia and a slight excess of neutral lead acetate solution are added, the mixture is boiled, again cooled, filtered, and the precipitate washed. The filtrate is acidified with acetic acid and boiled, sufficient "nitron" dissolved in acetic acid being added to precipitate all the nitrate present. The vessel containing the mixture is kept in ice-water for three hours, after which period the precipitate of "nitron" nitrate is collected on a filter, washed with 10 to 12 c.c. of ice-water, and dried at a temperature of 110° C. The weight of the precipitate multiplied by 0·26933 gives the amount of potassium nitrate in the 200 c.c. of solution taken for the determination. Basic lead acetate must not be used for clarifying the meat decoction, as its use tends to give low results; sodium chloride, even when present in the meat in considerable quantity, does not interfere with the determination, as most of it is removed with the lead precipitate. W. P. S.

The Determination of Unaltered Casein in Cheese. A. Trillat and Sauton. (*Ann. de Chim. Anal.*, 1906, vol. 11, 363-365.)—The unaltered casein in cheese forms an insoluble product with formaldehyde (ANALYST, xxxi., 260), whilst the broken-down products formed from the casein as the cheese matures remain soluble. Two grams of the sample are triturated with 10 c.c. of hot water, the mixture treated little by little with 50 c.c. more of water (very faintly ammoniacal water in the case of hard cheese), and boiled for five minutes. It is next treated with 0·5 c.c. of commercial formalin, boiled for three minutes longer, and allowed to stand for five minutes, for the fat to rise to the surface. The casein is then precipitated by means of 5 drops of pure acetic acid, and the precipitate collected, extracted with acetone, dried, and weighed as described in the previous communication. The following percentages of casein were thus obtained with commercial samples of cheese: Camembert, 18·20; Gruyère, 31·34; Gervais, 6·41; Brie, 22·93; Roquefort (half-ripe), 11·65; Roquefort (very ripe), 7·10; and Dutch, 31·5. The process of ripening may also be followed, and the ratio between the amounts of original and altered casein determined. Thus a sample of fresh Roquefort cheese gave the following results: Original cheese, 19·48; after eight days, 18·12; after fifteen days, 11·65; after thirty days, 8; and after sixty days, 7·10 per cent. of unaltered casein. The authors have proved by control experiments that the casein separated by this method does not contain any foreign substances, and that it has the composition of milk casein; also that any peptones or albumoses formed are not rendered insoluble by formaldehyde. They have obtained similar results in the artificial digestion of casein with pepsin. C. A. M.

On the Carbohydrates occurring in Spices. 1. In Canella Bark. J. Hanus and F. Bien. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 395-407.)—The quantity of pentosan occurring in spices is fairly constant for each kind of spice; the largest amount (18·28 per cent. on the dry substance) being found in canella bark and the least in those spices which consist of the flowers of the plant. The quantity of crude fibre yielded by a spice is proportional to the pentosan content. The pentosans found in canella bark are not completely hydrolysed by 8 per cent. sulphuric acid, a considerable portion, closely resembling cellulose, remaining unattacked. Canella bark contains about 8 per cent. of mannite, and, on treating the bark with water under pressure, araban, galactan, and small quantities of xylan go into solution. The residue from the aqueous extraction still contains xylan and, possibly, dextrosan, these two polysaccharides dissolving when the residue is treated with 5 per cent. sulphuric acid. W. P. S.

The Quantity of Tin in Tinned Tomatoes. C. Formenti and A. Scipiotti. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 290-293.)—From 0·091 to 0·128 per cent. of tin was found in six samples of tinned tomato pulp, the determinations being made immediately after the tins were opened. In two cases the pulp was left in the tins for five days, and the tin content of the pulp again determined. In one sample the quantity of tin increased from 0·091 to 0·324 per cent., and in the other from 0·093 to 0·169 per cent. As Ungar (*Zeit. für Hygiene*, 1887, ii., 241), Günther (*Rev. intern. falsif.*, 1887), and others have published results showing the injurious effect of tin compounds on animals, it is important that the dissolving action of fruit acids on tin should be prevented as far as possible, either by varnishing the interior of all tins in which fruits, etc., are preserved, or by removing the fruit from the tin as soon as the latter is opened. W. P. S.

Determination of Cellulose, Lignin, and Cutin in "Crude Fibre." J. König. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 385-395.)—Methods have already been given for the determination of cellulose and lignin in "crude fibre" (ANALYST, 1903, p. 318), and it is now shown that the "cellulose" thus obtained may be further split up, by treatment with ammoniacal copper solution, into pure cellulose and cutin. The "crude fibre" is determined as already described, and a second portion of the sample is treated according to the process given previously, that is, with glycerol and sulphuric acid, and afterwards with ammoniacal hydrogen peroxide. The residue obtained is, together with the asbestos filter, now treated for two hours with 75 c.c. of ammoniacal copper solution, prepared by saturating 24 per cent. ammonia with hydrated cupric oxide (Merck). The mixture is then gently warmed and filtered through asbestos, the residue is washed with water, dried on the filter at 110° C., and weighed. After ignition, the filter is re-weighed, the difference in the two weighings giving the weight of the cutin.

Three hundred c.c. of 80 per cent. alcohol are next added to the filtrate from the cutin residue; on stirring the mixture, the cellulose separates out and is collected on an asbestos filter, dried at 110° C., and weighed. The difference between the quantity of "crude fibre" and the sum of cellulose and cutin gives the amount of the oxidizable part of the "crude fibre," the so-called lignin. W. P. S.

Further Simplification of the Determination of "Pepsin-Soluble" Nitrogen in Feeding-Stuffs. A. Stutzer, H. Wangnick, and W. Rothe. (*Journ. Lanw.*, 1906, vol. 54, p. 265; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 338.)—A strong extract for determining the pepsin-soluble nitrogen in feeding-stuffs is prepared by cutting up the inner mucous coat of at least six pig stomachs and allowing the fragments to stand, with occasional agitation, in 0·2 per cent. hydrochloric acid for twenty-four hours in a cool place, 2·5 litres of liquid being taken for every stomach used. The extract obtained is filtered first through flannel and then through paper. So much chloroform is then added that a portion remains undissolved, and the liquid is kept in a cool place. A. G. L.

Determination of Digestible Proteids in Feeding-Stuffs. A. Stutzer. (*Journ. Landw.*, 1906, vol. 54, p. 235; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 338.)—The separation of proteids from non-proteids by means of copper hydroxide has been proved to work well, both when previously prepared copper hydroxide or when a mixture of copper sulphate and sodium hydroxide is used. In both cases the liquid should be kept slightly acid; this may be done by adding a few c.c. of strong alum solution, or by employing an excess of copper sulphate solution. To determine "pepsin-soluble" nitrogen, 2 grams of the feeding-stuff are digested for forty-eight hours at blood temperature with 500 c.c. of gastric juice prepared according to the author's old, or 250 c.c. according to his new, prescription (see preceding abstract). The amount of proteid actually digested by the animal stomach is, however, often different from that indicated by the quantity of "pepsin-soluble" nitrogen found. A. G. L.

Characteristics of Annam Beeswax. J. Bellier. (*Ann. de Chim. Anal.*, 1906, vol. 77, pp. 366-368.)—The sample of this wax examined by the author was in the form of prismatic cakes containing 5·02 per cent. of water, 0·5 per cent. of substances insoluble in benzene, and 0·08 per cent. of ash. The wax itself, when melted and filtered, resembled European waxes in appearance. It gave the following results on analysis: Specific gravity, 0·964; melting-point, 61° C.; acid value, 7·8; ester value, 86·6; ratio of acid value to ester value, 11; iodine value, 6; hydrogen liberated at 250° C. by potash-lime, 60·3 c.c. per gram at 0° C. and 760 mm.; and hydrocarbons unsaponifiable at 250° C., 10·5. The wax thus differs from European waxes in having a higher iodine value, and containing less free acids and more combined acids, so that the ratio is 11 instead of the usual 3·8. The amount of hydrogen liberated is also much greater. On the other hand, these constants are very similar to those of Indian waxes, and in particular of that of *Apis dorsata* (see ANALYST, xxx., 57). C. A. M.

The Characteristics of Essential Oil of Juniperus Phœnicea. J. Rodié. (*Bull. Soc. Chim.*, 1906, xxxv., 922-925).—This oil is stated to be sold in France in place of savin oil (*Juniperus sabinus*), which it closely resembles. The pharmaceutical and chemical differences were studied by Umney and Bennett (*Pharm. Journ.*, lxxv., 827), who found that the substitute had a specific gravity of 0·892 at 15° C. and an optical rotation of + 4° 30′ in a 100 mm. tube. Five samples of the oil of undoubted purity examined by the author had a specific gravity of 0·867 to 0·868 at 15° C., and an optical rotation of + 2° 54′ to + 4° 10′. The low specific gravity of some of these samples was attributed to there being a larger proportion of terpenes in oil from younger trees and differences in the mode of distillation. Umney and Bennett found only 64 per cent. of constituents boiling below 165° C., whilst a sample distilled by the author yielded 92·3 per cent. of a terpene fraction boiling between 154° and 180° C., nearly the whole passing over between 155° and 160° C. In addition to pinene, already identified by the English chemists, this fraction was found to contain traces of camphene and phellandrene. C. A. M.

Characteristics and Reactions of Oil of Cade. C. Pépin. (*Journ. Pharm. Chim.*, 1906, xxiv., 248-259.)—From an examination of samples of known purity the author concludes that genuine oil of cade derived from *Juniperus oxycedrus* should have the following characteristics: It should be fluid, have an unmistakable odour of smoke, and a specific gravity slightly less than that of water. Its acidity, in terms of acetic acid, should be less than 1·5 per cent. When distilled under the ordinary pressure it should yield at least 65 per cent. of distillate between 150° and 300° C., and at least 70 to 75 per cent. between 10° and 215° C., on distillation under a pressure of 0·065 mm. A sample of pine-tar oil had an acidity of 1·555 per cent., and gave 15·2 per cent. of distillate between 150° and 300° C. under the ordinary pressure, and 23·2 per cent. between 10° and 215° C. under the reduced pressure. Pure oil of cade should always give a brown coloration in the test for pine-tar oil with petroleum spirit and copper acetate (ANALYST, xxxi., 302). In the author's opinion, no reliance can be placed on the colour reactions given by Hirschsohn and Adam, or in the results of the tests for furfural or pyrocatechol, which, according to Kauffeisen, are present in pine-tar oil, but not in aqueous solutions of true oil of cade. All the samples examined by the author gave the reactions for both substances.

C. A. M.

Determination of Minute Quantities of Morphine. C. Mai and C. Rath. (*Archiv Pharm.*, ccxl., 300; through *Pharm. Journ.*, 1906, vol. lxxvii., p. 413.)—With a view of obtaining a means for determining quantities of morphine of less than 1 mgm. in weight, the authors have investigated the iodic acid reaction, the coloration with Fröhde's reagent and that with Marquis's reagent. The latter consists of a mixture of 2 drops of 40 per cent. formaldehyde solution with 3 c.c. of sulphuric acid. Of these reactions that with Marquis's reagent was the only one that proved to be practicable. It is carried out as follows: One c.c. of a 0·1 per cent. morphine hydrochloride solution is evaporated to dryness in a basin, and the residue mixed with 1 c.c. of the reagent. The liquid is transferred to a small test-tube, and the basin rinsed with 4 c.c. of sulphuric acid. With 0·001 gram of the alkaloid the mixture assumes a deep violet-blue colour; with smaller quantities the coloration is less intense and is easily comparable colorimetrically. The limit of morphine that can be determined is 0·00003 gram. W. P. S.

TOXICOLOGICAL ANALYSIS.

A Simple Method of Differentiating the Blood of Different Animals. Piorkowski. (*Ber. d. pharm. Ges.*, 1906, 226; through *Journ. Pharm. Chim.*, 1906, xxiv., 272, 273.)—The following simple method of applying the serum test for blood has been devised by the author: One c.c. of hydrocele liquid, ascites fluid, or of human serum, is introduced into each of a series of test-tubes (6 c.m. long by 0·8 c.m. in diameter), then one drop (0·04 c.c.) of the fresh blood of different animals diluted ten to fifty times, or better, of the dried blood previously dissolved in a physiological solution of salt. Hydrocele fluid forms the best reagent, and the blood must be allowed to fall from the pipette so as to form a layer on the surface of the other liquid. The tubes are examined thirty to forty-five minutes after the introduction of the blood, and if the latter is of human origin a faint red precipitate (of coagulated blood) will be observed, while the supernatant liquid will have remained clear. On the other hand, the blood of other species will have dissolved in the human serum, colouring it red. If the tubes are charged, in the first place, with serum of the horse, ox, or other animal, the corresponding blood is coagulated, while that of any other animal dissolves. The reactions are rendered more conclusive by cautiously shaking the tubes every half-hour after the coagulation has taken place, a fresh precipitate being formed each time. In preparing the solution of blood, the action of the sodium chloride (or sodium carbonate) upon the stain, etc., is continued until the liquid is manifestly yellow, after which it is filtered and tested as described.

C. A. M.

ORGANIC ANALYSIS.

Detection of Methyl Alcohol. H. Scudder and R. B. Riggs. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 1202-1204.)—Oxidation of the solution by a hot copper spiral, subsequently testing for formaldehyde by heating with milk and HCl containing Fe_2Cl_6, is unreliable for the detection of methyl alcohol, 10 per cent. aqueous solutions of ethyl alcohol, acetic acid, and acetone, all giving reactions indistinguishable from that obtained with pure methyl alcohol. The following modification of the Senglé-Ferrière-Cuniasse test shows 2 to 3 per cent. of methyl alcohol in ethyl alcohol. To 10 c.c. of the aqueous solution to be tested add 0·5 c.c. concentrated H_2SO_4, and 5 c.c. of a saturated solution of $KMnO_4$, the temperature being maintained at 20° to 25° C. After two minutes add sufficient sulphurous acid to give a colourless solution, boil till free from SO_2 or acetaldehyde, and apply the resorcinol test for formaldehyde. The production of a pink ring is usually sufficiently characteristic to prove the presence of methyl alcohol, but the appearance of flocks, which can often be developed by standing for one or two hours and then heating the upper layer to boiling, affords positive evidence of its presence. The use of HCl instead of H_2SO_4 in the resorcinol test prevents any darkening due to overheating or charring, but lessens the delicacy of the reaction. W. H. S.

The Formation of Formaldehyde in the Caramelization of Sugar.

A. Trillat. (*Bull. Soc. Chim.*, 1906, xxxv., 681-685.)—On heating sugar, traces of formaldehyde are given off even at 125° C., and at 150° C. the aldehyde is liberated in greater quantity. Analyses of the vapours emitted at 200° C. showed the presence of 0·2 to 5·7 per cent. of formaldehyde, and the residual caramel contained up to 0·27 per cent. of polymerized formaldehyde, probably in the state of trioxymethylene. The amounts produced were found to vary with the method of heating, the nature of the vessel, and the purity of the sugar. Experiments with five samples of commercial caramel showed that two were free from formaldehyde, whilst the others contained from 0·030 to 0·325 per cent. The author points out that this accounts for the antiseptic properties of caramel. Fresh urine, for example, treated with a sufficient quantity of caramel no longer putrefies. The *Bacillus coli communis* was destroyed by being left for fourteen hours in contact with a 10 per cent. solution of caramel. C. A. M.

The Determination of Formic Acid by Potassium Permanganate.

J. Klein. (*Ber.*, 1906, vol. 39, p. 2640; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 329.) —Grossmann and Aufrecht's recent paper on this subject has caused the author to refer to his method, published nearly twenty years ago. It consists in oxidizing formic acid in a boiling alkaline solution by means of potassium permanganate, then adding an excess of oxalic acid and dilute sulphuric acid, and finally titrating the excess of oxalic acid with permanganate. A. G. L.

On the Determination of Malic Acid. H. Cantoni and M. Basadonna.

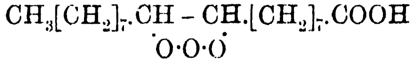
(*Bull. Soc. Chim.*, 1906, xxxv., 727-737.)—The determination of malic acid by oxidation with potassium permanganate in acid solution has yielded absolutely discordant results in the authors' hands, whatever modification of the method was employed. They have also obtained bad results with the various methods of precipitating the acid by means of lead acetate, the highest yield being 91·44 per cent. of the theoretical amount. In their opinion, there is as yet no method capable of determining malic acid in the presence of other organic acids, and they recommend the study of the solubility of malates in different solvents to find the basis for a reliable process. They themselves have determined the solubility of malates of the alkaline earths in water. The strontium salt is less soluble at 20° to 30° C., but much more soluble at higher temperatures than the calcium and barium salts, and its solubility increases enormously with the rise in the temperature. Calcium malate is the least soluble of the three salts at 35° C., and its solubility decreases with the rise in temperature. The barium salt is slightly more soluble in hot than in cold water (*cf.* ANALYST, xxxi., 300). C. A. M.

On Melezitose and Turanose. G. Tanret. (*Bull. Soc. Chim.*, 1906, xxxv., 816-825.)—The melezitose prepared by the author from Taschkent manna had a composition corresponding with the formula $C_{18}H_{32}O_{16}$, $2H_2O$, and did not lose the whole of its water of crystallization at 130° to 135° C. Its optical rotation, calculated on the anhydrous sugar, was $\alpha_D = +89 \cdot 3$. When hydrolysed by means of a dilute acid, it yielded one molecule of turanose and one molecule of dextrose, the former being decomposed on further hydrolysis into one molecule of dextrose and one molecule of lævulose, and not, as commonly accepted, into two molecules of dextrose. The turanose separated from the products of the restricted hydrolysis of melezitose had the formula $C_{12}H_{22}O_{11}$, and was the first known isomer of cane-sugar. It was very sweet and soluble in all proportions in water and methyl alcohol. Its optical rotation was $\alpha_D = +71 \cdot 8$ (calculated 72·8). It was fermented by beer-yeast, but only very slowly. From 5 to 10 per cent. of sugar remained unfermented in an experiment that was continued for five months. C. A. M.

Determination of Lead Number of Maple Syrup and Maple Sugar. A. L. Winton and J. L. Kreider. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 1204-1209.)—The proportion of lead in the precipitate produced by treatment of maple products with basic lead acetate solution is approximately constant, and furnishes a means of determining the presence of cane-sugar. For its estimation the authors recommend an indirect method, using a definite volume of standard lead subacetate, and determining the lead remaining in the solution after precipitation. Twenty-five grams of the material is weighed into a 100 c.c. flask, 25 c.c. lead subacetate solution added, and the volume made up with water to 100 c.c., the mixture shaken, allowed to stand one hour, and filtered. Ten c.c. of the clear filtrate are diluted to 50 c.c., a moderate excess of H_2SO_4 and 100 c.c. of 95 per cent. alcohol added, and the whole allowed to stand overnight. The precipitate is then filtered on a Gooch crucible, washed with 95 per cent. alcohol, dried at a moderate heat, ignited at low redness for three minutes and weighed, the difference between the amount of lead thus found and that present in 2·5 c.c. of the standard solution, when divided by 2·5, giving the "*lead number*." The lead subacetate solution used is prepared by boiling for half an hour 430 grams lead acetate, and 130 grams litharge, with 1,000 c.c. water, and after cooling and allowing to settle, diluting the supernatant liquid to a density of 1·25. To a measured volume of this solution, 4 volumes of water are added, filtering if not perfectly clear. The lead number for maple syrup is found to vary from 1·19 to 1·77, the last figure being obtained with a poor quality, and that for maple sugar from 1·84 to 2·48, falling in the case of adulterated samples to as low as 0·10, and in one case 0·02. W. H. S.

On a Method for the Separation of Cholesterol and Phytosterol. A. Windaus. (*Chem. Ztg.*, 1906, xxx., 1011.)—Cholesterol dibromide is difficultly soluble in a mixture of glacial acetic acid and ether, a mixture of 50 c.c. of acetic acid and 50 c.c. ether dissolving only 0·6 gram of the substance at 20° C. Phytosterol dibromide, on the other hand, is much more readily soluble, and, moreover, does not crystallize at all easily. The author proposes a method of separation based on these facts, and describes the treatment of mixtures of 4, 8 and 0·4 gram cholesterol with 4, 0·8 and 4 grams respectively of phytosterol, the mixtures being dissolved in each case in 10 parts of ether and treated with the same quantity of glacial acetic acid containing 5 per cent. of bromine. In each case products were obtained which permitted of ready identification as cholesterol and phytosterol derivatives. The dibromides can be readily reconverted into the original alcohols by treatment with a reducing agent (zinc dust or sodium amalgam). A. G. L.

The Action of Ozone on Oils and Fats. E. Molinari and E. Soncini. (*Berichte*, 1906, xxxix., 2735-2744.)—Ozone is absorbed quantitatively by unsaturated fatty acids, and the increase in weight corresponds with the ozone value calculated from the iodine value. Oils become very viscous under the action of the gas, and a good solvent for modifying the violence of the reaction has been found in hexane from petroleum, which can subsequently be easily eliminated by means of a current of ozone. The author has determined the ozone values of linseed, maize, castor, olive, and rape oils, etc., and has obtained results agreeing well with the iodine values. Linseed-oil, for instance, with an iodine value of 171 gave an ozone value of 30 as against the theoretical 32·3. Pure oleic acid (iodine value 90·03) absorbed ozone in the proportion of 1 molecule to 1 molecule (O_3). The ozonide was a viscous, almost colourless liquid, heavier than water, and decomposing without melting when heated above 90° C. Its elementary composition and molecular weight corresponded with the formula $C_{18}H_{34}O_5$. When decomposed by dry or moist heat it yielded nonylic, azelaic, and two other fatty acids, and an oily product boiling at about 190° C. From a study of these decomposition products the authors conclude that the ozonide must have the constitutional formula—



$$CH_3[CH_2]_7.\underset{\cdot O\cdot O\cdot O\cdot}{CH - CH}.[CH_2]_7.COOH$$

and that oleic acid absorbs 3 atoms of oxygen when acted upon by ozone, and not 4 as asserted by Harries (*Berichte*, xxxviii., 1630).

New Method of Determining Ozone.—When ozone is passed through ordinary olein the increase in weight corresponds to the amount of the gas absorbed.

C. A. M.

On the Ozonides of Oleic Acid. C. Harries and C. Thieme. (*Berichte*, 1906, xxxix., 2844-2846.)—In a previous communication (*Ann. d. Chem.*, 1906, 318) it was found by the authors that when ozone acted upon a solution of oleic acid in acetic acid or chloroform it yielded a viscous oil, which, when purified by being dissolved in acetic ether and precipitation by petroleum spirit, was a colourless product containing 4 atoms of oxygen. They now find that if this substance be washed with water and sodium bicarbonate solution and extracted with ether, it gives a product containing only 3 atoms of oxygen. They propose to term this normal oleic acid ozonide, and their former preparation oleic acid perozonide. Both ozonides yield the same products when decomposed by water, and the chief difference between them is that when equal quantities are boiled for the same length of time with water a much more intense reaction for hydroxgen peroxide is obtained in the case of the perozonide. The formula assigned by the author to the normal ozonide is the same as that given by Molinari and Soncini (see preceding abstract).

C. A. M.

Notes on some Conifer Oils. R. E. Hanson and E. N. Babcock. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 1198-1201.)—Several oils distilled by the authors from authentic materials have been examined with the following results:

	Yield. Per Cent.	Specific Gravity.	Ester. as Bornyl Acetate. Per Cent.	
Oil of Black Spruce	0·57	0·9274 at 19°	—	—
,, Hemlock (1)	0·4	0·9238 ,, 15°	—	—
,, ,, (2)	0·46	0·9273 ,, 15°	—	—
,, Cat Spruce, leaves	0·103	0·9216 ,, 15°	25·7	Odour suggests limonene or dipentene
,, Cat Spruce, cones	0·25	0·899 ,, 15°	—	Yellow colour, pronounced limonene-like odour
,, Red Spruce, leaves	0·204	0·9539 ,, 16°	66·2	Free borneol, 7·76 per cent. odour of bornyl acetate
,, Red Spruce, cones	0·38	0·8600 ,, 15°	—	Golden-yellow colour, fir-balsam-like odour
,, American Larch	0·149	0·8816 ,, 15°	15·1	Remainder largely pinene
,, Pitch Pine	0·001	—	—	Yellow colour, pungent odour
,, Red Pine	0·001	—	—	Brownish - red colour, pungent and disagreeable odour
,, Juniper, leaves	0·18	0·8531 at 20°	—	Light yellow colour, juniper odour
,, Virginia Juniper, leaves	—	0·900 ,, 16°		—

W. H. S.

The Phosphomolybdic Acid (Welman's) Test for Vegetable Oils. B. Kühn and G. Halfpaap. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, xii., 449-455.)—This test is quite useless for detecting the presence of vegetable oils in oleo-margarine and in tallow, as these two fats themselves give a dark green coloration with the reagent, but it may be employed for the detection of vegetable oil in lard. With very few exceptions, lards do not give a coloration. The constituent of vegetable oils which gives the coloration is, apparently, a decomposition product of albumin; it is non-volatile, easily oxidizable, and is readily removed from the oils by extraction with alcohol, but not with hydrochloric acid. Owing to the fact that this active constituent is destroyed by heat, direct sunlight and oxidizing agents, a negative reaction does not prove the absence of vegetable oils (*cf.* ANALYST, 1895, p. 62).

W. P. S.

The Analysis of Turkey-red Oils. W. Herbig. (*Chem. Rev. Fett-u. Harz-Ind.*, 1906, xiii., 187-190, 211-213, 241-244).—There is considerable confusion in commerce as to the meaning of the term "50 per cent. Turkey-red oil," etc., when applied to these products, and to obviate this the author suggests that it shall invariably be taken to refer to the amount of total fat. He has made comparative experiments with different methods, and recommends the following simple method of determining the total fat and combined sulphuric acid: Ten grams of the sample are heated with 50 c.c. of water, and the resulting solution treated with 25 c.c. of dilute hydrochloric acid (strength not given), and boiled for three to five minutes. When cold it is transferred to a separating funnel, the flask being rinsed out with water and ether, and then shaken with about 200 c.c. of ether. The aqueous layer is drawn off, and the ethereal solution thoroughly washed three times with water, which will take from thirty minutes to an hour. The washings are added to the original aqueous layer, the mixture boiled to expel all ether, and the sulphuric acid precipitated in the usual way as barium sulphate. The ethereal solution is now concentrated by distillation at a low temperature, the residue then transferred to a weighed beaker, and the rest of the ether allowed to evaporate spontaneously. Finally the fat is heated for a minute or so over a free flame (till no more bubbles appear), and is then dried for thirty minutes at 105° C., and weighed. Tables of experimental results are given in detail, and it is also shown that the results for the total fat obtained by decomposing Turkey-red oils with cold hydrochloric acid differ but slightly from those obtained by treating the oils with boiling acid. C. A. M.

A Method of Distinguishing between True Albumin and Mucinoid Substance in Urine. L. Grimbert and E. Dufau. (*Journ. Pharm. Chim.*, 1906, xxiv., 193-199.)—A little of the filtered urine is allowed to run from a narrow pipette on to a few c.c. of a solution of 100 grams of citric acid in 75 c.c. of water. When only mucinoid substance is present, a more or less nebulous zone is formed near the juncture of the liquids, attaining its maximum intensity after the lapse of one or two minutes. On the other hand, urine containing only pathological albumin (even as much as 6 to 8 grams per litre) gives no such turbidity. Again, when the urine is allowed to flow on to the surface of nitric acid (Heller's reaction), pathological urine produces a sharp ring at the contact-zone, whilst mucinoid substance gives no ring, but a turbidity above the zone of contact. In the case of urine containing both substances, the ring and turbidity will both appear, with a clear zone between them. When Heller's reaction gives a positive result, it will be necessary to confirm by other tests the nature of the proteid found.

C. A. M.

Note on the Analysis of Hungarian Leather. F. Jean. (*Ann. de Chim. Anal.*, 1906, vol. 77, pp. 361-363.)—The commissariat of the French Army requires the white skins intended for military accoutrements to contain not more than 10 per cent. of water, from 10 to 15 per cent. of alum, 5 per cent. of sodium chloride, and 25 to 33 per cent. of grease. By "alum" is understood potassium aluminium sulphate containing twenty-four equivalents of water. Three samples of such leather examined by the author gave the following results: Water (loss at 105° C.), 25·4 to 26·2; fat, 21·5 to 23·6; sodium chloride, 4·1 to 5·6; sulphuric acid (SO_3), 6·7 to 9·3; water in ammonium sulphate, 0·25 to 0·31; ammonia, 0·47 to 0·49; alumina, 2·4 to 3·9, and hide substance (N × 5·62), 32·7 to 36·3 per cent. Calculation of the amount of ammonium alum (containing twenty-four equivalents of water): (1) from the amount of alumina; (2) from the total sulphuric acid; and (3) from the ammonia, gave the following figures:

	1 Per Cent.	2 Per Cent.	3 Per Cent.
From the alumina	21·5	26·7	34
„ sulphuric acid	19·19	19·54	26·29
„ ammonia	12·78	12·60	15·77

The author's explanation of these discordant results is that in hides whitened with alum and salt the aluminium is no longer present in the form of alum, but that there has been dissociation of the salt into a more or less basic aluminium sulphate fixed by the hide, with partial elimination of ammonium sulphate in the baths. It will be necessary for the military specifications to be modified, since ordinary alum has now been generally replaced by ammonium or sodium alums in the preparation of Hungarian white skins.

C. A. M.

On the Balsam of Hardwickia Pinnata. G. Weigel. (*Pharm. Centralh.*, 1906, vol. 47, p. 773; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 345.)—This East Indian balsam appears capable of replacing copaiva balsam in the painting of porcelain, varnish-making, etc. The balsam contains 48·5 per cent. of ethereal oil and 51·5 per cent. of resinous bodies (48·3 per cent. saponifiable and 3·2 per cent. unsaponifiable).

A. G. L.

The Determination of the Calorific Value of Liquid Fuels by Means of the Junker Calorimeter. E. Glinzer. (*Zeits. angew. Chem.*, 1906, xix., 1422.)—The author mentions the excellent results obtained by means of the Junker calorimeter in the case of gaseous fuels, and its increasing application to liquid combustibles. Under special conditions oils with a boiling-point of even 400° C. may be burnt in it, and in the ordinary way the calorimeter may be used for oils with a boiling-point not exceeding 250° C. For such oils the burner shown in the figure is used, the principle of the burner being similar to that of the well-known "Primus" burners for gasolene and petroleum. The burner is suspended from one arm of a balance, and nearly balanced by weights placed on a scale-pan depending from the other arm. The oil-vapour is then lighted, the nozzle placed in the calorimeter, the water is turned on, and the temperatures of the in-going and out-coming water are allowed to become constant. When so much oil has been burnt that the burner is exactly balanced, a small weight (10 or 20 grams) is placed in a small scale-pan below the burner so as to depress the latter, and the experiment is continued until the burner is again in exact equilibrium, showing that a quantity of oil corresponding to the added weight has been burnt. The water passing through the calorimeter during this time is, of course, collected and measured. A. G. L.

INORGANIC ANALYSIS.

A Delicate Colour Reaction for Copper, and a Micro-chemical Test for Zinc. Harold C. Bradley. (*Amer. Journ. Sci.*, xxii., No. 130; through *Chem. News*, 1906, vol. 94, p. 189.)—It has long been known that hæmatoxylin (the unoxidized extract of logwood) gives a dark blue colour with copper salts. The author has found that the reaction is exceedingly delicate, solutions containing only 0·0000001 per cent. of copper still giving a blue colour, whilst the limit with ferrocyanide is 0·001 and with potassium iodide 0·0001 per cent. The absolute quantities taken are not stated.

Zinc can be readily identified in the presence of other bodies by its characteristically crystalline nitro-prusside, all the other heavy metals giving amorphous, slimy nitro-prussides. Thus, zinc was recognised in the blood of certain molluscs by incinerating a small quantity, preparing a roughly 10 per cent. solution of the ash, removing copper by means of sulphuretted hydrogen, evaporating the filtrate to a small bulk, and digesting 1 drop of the solution on a microscope slide with 1 drop of a fresh solution of nitro-prusside. On cooling, the rectangular plates and prisms of zinc nitro-prusside could be readily seen under the microscope. A. G. L.

Estimation of Copper by Titanium Chloride. Ezra Lobb Rhead. (*Journ. Chem. Soc.*, 1906, vol. lxxxix., p. 1491.)—In presence of potassium thiocyanate, copper is quantitatively reduced from the cupric to the cuprous state by a solution of titanium trichloride. The reaction takes place in either sulphuric acid or hydrochloric acid solutions. As indicator a little ferrous sulphate is added, which is oxidized by the cupric salt to the ferric salt, the latter giving the well-known red colour of ferric thiocyanate to the solution; the disappearance of this colour marks the end of the reaction. The titration must be carried out below 30° C. and in an atmosphere of hydrogen or other inert gas, as titanium trichloride oxidizes readily in air. Ferric salts must be absent or else their amount known and allowed for; nitric acid must also be absent. The results given by the method are good. A. G. L.

The Determination of Arsenic Acid. L. Rosenthaler. (*Zeit. anal. Chem.*, 1906, xlv., 596-599.)—The method is based on the following reaction (the converse of that in general use in the determination of arsenious acid):

$$2H_3AsO_4 + 4KI + 4HCl = As_2O_3 + 4I + 4KCl + 5H_2O.$$

In the presence of a large excess of acid, the reaction begins without the application of heat, and is complete in ten to fifteen minutes, after which the liberated iodine can be titrated with $\frac{N}{10}$ thiosulphate solution, each c.c. of which corresponds to 9 mgms. of potassium arsenate. In making a determination, the potassium arsenate is dissolved in water, 2 grams of potassium iodide introduced, and hydrochloric acid (25 per cent. strength) added until a precipitate results. This is dissolved by the addition of the smallest possible amount of water, and the liquid allowed to stand ten to fifteen minutes before titration of the iodine. Sulphuric acid can be used in place of hydrochloric acid with equally satisfactory results.

For the simultaneous determination of arsenic and arsenious acids, the latter is first titrated in the usual way with iodine solution. The liquid is then treated with 1 to 2 grams of potassium iodide, and fuming hydrochloric acid and 50 per cent. sulphuric acid (proportions not given) introduced until the precipitate forms, after which the determination is completed as above described. The method can also be used in the determination of arsenic, the latter being first oxidized into arsenic acid.

C. A. M.

Determination of Minute Quantities of Arsenic. W. Thomson. (*Chem. News*, 1906, vol. xciv., pp. 156, 157, and 166, 167.)—The author points out that all nitrous compounds formed in the destruction of organic matter must be completely removed by repeated evaporation in order to obtain the proper size and depth of the arsenic mirror when the electrolytic process is employed for the determination. With the Marsh-Berzelius method the presence of some nitrous compounds does not materially affect the results. Results of experiments are also given to show that lead cathodes are the most efficient in reducing arsenic compounds, in the presence of nitric or nitrous acid, to the state of arseniuretted hydrogen. From experiments with insensitive zinc in the Marsh-Berzelius process the author concludes that the addition of cadmium sulphate does not always have the effect ascribed to it by Chapman and Law (ANALYST, 1906, p. 3).

W. P. S.

Investigations on Red Phosphorus. A. Siemens. (*Arb. aus d. Kais. Gesundheitsamte;* through *Chem. Ztg.*, 1906, xxx., *Rep.*, 349.)—The author shows that every sample of red phosphorus can be made to give indications of the presence of yellow phosphorus in the Mitscherlich test, if only the boiling is sufficiently rapid (under somewhat reduced pressure), and a current of hot oxygen is employed. The "glowing" of phosphorus appears to be due to the formation of a volatile lower oxide, which is formed from red phosphorus at 90° C. at the same rate as from yellow phosphorus in the cold. The author has worked out a method for the detection of yellow in red phosphorus, which depends on the reduction to metal of certain metallic salt solutions by means of the dissolved phosphorus. He also shows the view that concussion changes red into yellow phosphorus to be untenable; the red phosphorus is only changed into a more finely divided modification, in which state it is more easily acted on by reagents and more readily soluble.

A. G. L.

The Detection of Ozone. F. Fischer and H. Marx. (*Berichte*, 1906, xxxix., 2555-2557.)—Paper dipped in an alcoholic solution of tetramethyl-*p*-diamido-diphenylamine becomes violet when brought in a moist condition into contact with ozone. Oxides of nitrogen colour this "tetramethyl base" paper a straw yellow, whilst mixtures of nitrous oxides and ozone give dirty-brown intermediate shades. It is essential to have the test-paper moist, since the dry paper is coloured yellow by the long-continued (thirty minutes) action of ozone. If either the nitrous oxide or the ozone largely predominates in a mixture of the gases, the paper gives the colour characteristic of the gas that is in excess. Paper that is coloured violet by ozone becomes yellow if subsequently exposed to the action of nitrous oxides, and *vice versâ*. A trace of nitric oxide in ozone may be detected by conducting the mixed gases into liquid air, when the ozone dissolves and the nitric oxide solidifies in flakes which can be separated by filtration, while the ozone remains in the filtrate. C. A. M.

The Examination of Liquid Carbon Dioxide. Werder. (*Chem. Ztg.*, 1906, xxx., 1021.)—According to the author, the spread of the total abstinence movement has led to a considerable increase in the quantity of liquid carbon dioxide used in the manufacture of aerated waters and fruit syrups. The quantity of liquid carbon dioxide made in Switzerland is estimated for the year 1906 at 300,000 to 350,000 kilogms., the greater part being for human consumption. The carbon dioxide is made in three ways: from coke, potassium bicarbonate being an intermediate product; by heating magnesite or other carbonates; and from breweries.

The author proposes the following specification for the liquid: There should be no pungent or empyreumatic odour. The taste should be purely acid. The quantity of carbon dioxide present should not be less than 98 per cent., that of carbon monoxide not more than 0·5 per cent. Sulphurous and nitrous acids must be absent. On leading the gas for fifteen minutes through 100 c.c. of a warm $\frac{N}{100}$ potassium permanganate solution containing sulphuric acid, no appreciable decolorization should take place; nor should it produce a precipitate in the same time in 100 c.c. of a $\frac{N}{100}$ silver nitrate solution acidified with nitric acid.

In taking a sample of the gas, the bottle should always be in the horizontal position; otherwise the quantity of air in the sample taken is excessive. The carbon dioxide, oxygen, and carbon monoxide present are best determined in an Orsat apparatus, the measuring vessel of which is narrowed in its upper part, so that 20 c.c. can be read to $\frac{1}{10}$ c.c. The measuring vessel is filled ten or twenty times with the gas, the carbon dioxide being absorbed each time, and the united residues are then analysed for oxygen (0·01 to 0·05 per cent.) and carbon monoxide (0·05 to 0·20 per cent.). Communication between the Orsat apparatus and the reducing valve of the bottle is conveniently made by a tube carrying a three-way tap. A. G. L.

Determination of Nitrates. Frank Sturdy Sinnatt. (*Proc. Chem. Soc.*, 1906, vol. 22, p. 255.)—Knecht and Hibbert's method for the determination of picric acid (*Ber.*, 1903, xxxvi., 1549) was applied to the determination of potassium nitrate as follows: Ten c.c. of a 0·1 per cent. solution of potassium nitrate were evaporated to dryness, and the residue heated for thirty minutes in a steam-oven with 5 c.c. of a solution of phenol-sulphonic acid. The whole was then washed into a flask, hydrochloric acid added, and titrated with titanium trichloride. Three titrations carried out in this way gave 0·009788, 0·009948, and 0·009817 gram of potassium nitrate respectively, instead of the 0·01 gram taken. A. G. L.

The Determination of Hydrosulphurous Acid in Hydrosulphites and in their Combinations with Formaldehyde. A. Seyewetz and Bloch. (*Bull. Soc. Chim.*, 1906, xxxv., 293-297.)—The method is based upon the fact that hydrosulphurous acids and hydrosulphites reduce silver halides to metallic silver. The hydrosulphite is dissolved in boiled distilled water, with precautions to exclude atmospheric oxygen, and is immediately introduced into an ammoniacal solution of silver chloride containing four or five times the theoretical amount of silver salt for the reduction. The precipitated silver is collected, washed with ammoniacal water, ignited, and weighed. The reaction may be expressed by the equation :*

$$2AgCl + 4NH_3 + Na_2S_2O_4 + H_2O = 2NaCl + 2(NH_4)_2SO_3 + Ag_2.$$

The results quoted show that the method gives better results than those obtained by titration with indigo carmine.

The commercial product hyraldite (a mixture in varying proportions of sodium hydrosulphite-formaldehyde and sodium bisulphite-formaldehyde) cannot be titrated with indigo carmine. In aqueous solution it only reduces ammoniacal silver chloride slowly, but the reduction is immediate and complete at 80° C. The solution of hyraldite should be boiled for four or five minutes with four times the required amount of the reagent, and the precipitated silver weighed as in the case of hydrosulphites. Assuming that a molecule of pure hyraldite ($NaHSO_2 + CH_2O + 2H_2O$) reduces 2 molecules of silver chloride, the commercial hyraldite, C, of the Lyons manufacturers contains from 84·42 to 86 per cent. of the pure compound. The formaldehyde has no action upon the ammoniacal silver chloride, and is itself transformed in the reaction into hexamethylene-tetramine. C. A. M.

The Action of Sulphides on Nitroprussides. J. F. Virgili. (*Zeit. anal. Chem.*, 1906, xlv., 409-439.)—A soluble sulphide acting on sodium nitroprusside, or, in general, on any soluble or insoluble nitroprusside, yields a blue substance, which the author concludes to be a molecular addition compound. The presence of free alkali or of alkaline earths prevents the formation of this blue coloration, and certain salts of weak acids which can yield alkalies on hydrolysis—*e.g.*, silicates, phosphates, and borates—also interfere with the reaction. Ammonia and its salts have less influence. If an excess of a soluble nitroprusside act upon a soluble sulphide in the presence of a sufficient quantity of free alkali, a red coloration is obtained. The author attributes the reddish-yellow, red, purple and violet colorations that are frequently obtained to the simultaneous production of the blue compound between the sulphide and nitroprusside, and of a yellow substance formed by the action of alkali or alkaline earth upon the nitroprusside. As regards the use of nitroprussides in analysis, the conclusion is arrived at that they are not reagents for the sulphide ion, but for the un ionized sulphide molecule, and are hence less sensitive than the solutions of certain metallic salts—*e.g.*, neutral or alkaline lead salts—which act upon the sulphide ion. The sensitiveness of sodium nitroprusside as a reagent for sulphides is increased by the use of solvents—*e.g.*, glycerin or alcohol—which prevent or check the ionization, and also by the addition of an excess of the reagent, or by the intro-

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Assuming that, in the indigo process, one molecule (?) $Na_2S_2O_4 + 2H_2O$ (formula of Bernthsen) duction of other ions, for which purpose neutral salts, and in particular alkali carbonates, are suitable. The addition of alkali greatly increases the sensitiveness of the test, fixed alkalies being more effective than ammonia. If the sulphide solution containing the nitroprusside, but not an excess of alkali, be cooled to the freezing-point, the sensitiveness of the reaction is almost doubled, but even then falls short of that shown by reagents acting directly upon the sulphide ion. The maximum of sensitiveness is reached with an excess of alkali in the frozen solution, but the presence of the alkali prevents a distinction being made between sulphides and hydrogen sulphide. It is not possible to differentiate sulphides from sulphydrates by means of the nitroprusside reaction ; nor can a colorimetric determination of sulphides be made, owing to the difficulty of obtaining comparable coloured solutions and the want of sensitiveness of the reagent. C. A. M.

The Determination of Halogen. James Moir. (*Proc. Chem. Soc.*, 1906, vol. 22, p. 261.)—The substance to be analysed is weighed into a tall nickel crucible, 10 drops of water and 5 to 7 " pastilles " of pure potassium hydrate are added, and the mixture stirred with a platinum wire whilst it is heated on a steam-bath ; when it is uniform, 0·5 to 1 gram of finely-powdered potassium permanganate is gradually stirred in ; the heating is then continued until the mixture is dry, care being used to prevent frothing ; the crucible is next heated to redness over a burner, until the reaction between carbon and manganese dioxide is over. The cool crucible is next placed in a warm dilute solution of potassium bisulphite ; the solution obtained is acidified with acetic acid, and filtered into a solution of silver nitrate, the silver halide precipitated being treated as usual. Or the contents of the crucible may be dissolved in water, and acetic acid added until the manganate has been converted into permanganate ; the latter is destroyed by adding barium dioxide, and the filtered solution is neutralized with sodium bicarbonate and titrated, using chromate as an indicator. A. G. L.

Modification of the Hanging-Drop Fluoride Test. C. D. Howard. (*Journ. Amer. Chem. Soc.*, 1906, xxviii., 1238, 1239.)—A test-tube of small bore, about 2 inches long, is fitted with a rubber stopper, into the bottom of which passes a small piece of glass-tubing closed at one end, its open end extending about 3 mm. into the tube. The precipitate of $CaCO_3$ and CaF_2, ignited until nearly free from carbonate, is well mixed with about 0·1 gram of precipitated silica, and introduced into the dry test-tube, the glass tubing nearly filled with two or three drops of water, the bottom of the stopper thoroughly dried, and the stopper inserted immediately after the addition of 1 to 2 c.c. strong H_2SO_4. The tube is then immersed in a beaker of boiling water for fifteen to thirty minutes, when, if the solution under examination contains any appreciable quantity of fluoride, a heavy gelatinous ring will be formed in the small tube projecting from the stopper. By comparison with tubes containing known amounts of fluoride, the process may be made roughly quantitative. W. H. S.

Back Reactions in Iodine Titrations. J. H. Davies and E. P. Perman. (*Chem. News*, 1906, 93, p. 225.)—The " back reaction " often observed in the titration of iodine liberated from potassium iodide, using starch as indicator, is found to depend on the concentration of the potassium iodide in solution, and is most marked when the iodine is liberated by copper sulphate or potassium bichromate, but only very slight when bleaching-powder or permanganate is used. It appears to be due to the comparative slowness and incompleteness of the liberation of iodine, and may be overcome by increasing the concentration of the iodide solution to 1 gram in 10 c.c. water for 25 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ and 2 grams in 20 c.c. water for 1 gram of $CuSO_4 5H_2O$ in 50 c.c. water. W. H. S.

The Separation of Silica in the Determination of Citric-Acid-Soluble Phosphoric Acid. J. Hasenbäumer. (*Chem. Zeit.*, 1906, xxx., 665, 666).—The author finds that the magnesium pyrophosphate obtained in determinations of the citric-acid-soluble phosphoric acid in Thomas slag does not contain silica, and consequently the high results found when the silica has not been separated previously are not due to occluded silica. Further experiments make it appear probable that the higher results obtained when the silica has not been separated are due in part to the possible retention of phosphoric acid by the gelatinous silica, and also to the fact that the magnesium-ammonium phosphate has a slightly varying composition, depending on the previous separation of the silica. The ratio of magnesia to phosphoric acid is 1 : 2·02 when the silica has not been separated previously, and 1 : 1·95 in cases where the silica has been removed. W. P. S.

New Method for Preparation of Standard Solutions. S. F. Acree and R. F. Brunel. (*Amer. Chem. Journ.*, 1906, vol. 36, pp. 117-123.)—For the preparation of solutions of HCl and NH_4OH the authors recommend passing the purified and well-dried gases into a tared graduated flask, nearly filled with conductivity water, until the necessary weight is dissolved, the solution being then made up to the mark with water. The titration of solutions of HCl and H_2SO_4 with recrystallized $NaHCO_3$, weighing the resultant NaCl or Na_2SO_4, is considered preferable to the silver chloride and barium sulphate methods, while the unknown strengths of an acid and of an alkaline solution can be determined simultaneously by neutralizing one with the other and evaporating to dryness, the weight of the dry salt and the volumes of acid and alkali solutions used furnishing the necessary data.

W. H. S.

On Nessler's Reaction. A. Buisson. (*Journ. Pharm. Chim.*, 1906, xxiv., 289-294.)—It is shown experimentally that the reaction of ammonia with Nessler's reagent is incomplete, and that a state of equilibrium is established between the different substances. The ammonia is not completely precipitated, and on filtering off the precipitate and distilling the filtrate, the distillate will give a fresh reaction with Nessler's solution. Thus, in one experiment in which a solution containing 0·189 gram of ammonium chloride in 40 litres was treated in this way, the distillate contained 0·040 gram of ammonia, representing 21 per cent. upon which the reagent had not acted in the first instance. The author also finds that the precipitate has not the composition usually attributed to it. The simplest formula corresponding with his analytical results is $Hg_9N_4I_6$. The compound is a brown amorphous body, insoluble in neutral solvents, but soluble in excess of potassium iodide solution, which liberates the whole of its nitrogen in the form of ammonia—$HgN_4I_6 + 12KI + 12H_2O = 9HgI_2 + 4NH_3 + 12KOH$. In the colorimetric method only a portion of the ammonia contributes to the formation of the colour, and, in the author's opinion, it is probable that the intensity of the reaction in the standard solution and in the unknown solution is liable to vary in different fashion under the influence of different factors that affect the equilibrium, such as heat, dilution, etc. C. A. M.

The Differentiation of Natural and Artificial Mineral Waters. D. Negreano. (*Comptes Rendus*, 1906, cxliii., 257, 258.)—The electrical resistance of a natural mineral water is almost a physical constant for each kind of water, and serves to distinguish it from other waters. Thus the author obtained the following results in ohms—c.c. at 18° C.: Caciulata water (Roumania), 328; Slanic water, 48; Vichy water, 140; Vittel water, 500; and Évian water, 1,280. In determining the resistance at different temperatures, it was found that it diminished with the temperature. When the interval between the temperatures was not too great the resistance R_t at a temperature t could be calculated to 18° C. by means of the formula—

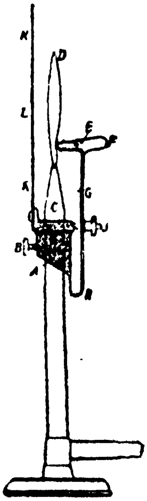
$$R_t = R_{18}[1 - a(t - 18)],$$

where a represents the coefficient of variation, and is approximately 0·02. The values of a in the case of waters mentioned above ranged from 0·019 to 0·027. The resistance of an artificial water made up to imitate the natural mineral water differs materially from that of the latter at the same temperature. For instance, water from the Célestine spring at Vichy showed a resistance of 140 ohms at 18° C., as against 112 ohms by an artificial Vichy water. The resistance of water of natural Évian water was 1,280 ohms, whilst that of an artificial water was only 1,120 ohms at 18° C. C. A. M.

APPARATUS.

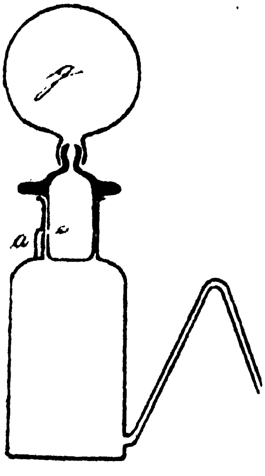
On a Petroleum Coke for Metallurgical Laboratory Use. F. O. Doeltz. (*Chem. Ztg.*, 1906, xxx., 585.)—The Celle-Wietze Petroleum Company in Hanover are supplying a petroleum coke well suited for many uses in the laboratory and for the manufacture of electrodes. It forms a pitch-black, shining, porous mass of considerable mechanical strength. Its calorific value was determined at 7,330 calories; its ash content varies from 0·21 to 0·53 per cent. The ash shows a tendency to fuse from the presence of alkali. The production in the year 1905 was 55,000 kilogms. of the coke, its present price being 5 marks per 100 kilogms. A. G. L.

A New Method for the Standardization of Thermometers below 0° C. T. W. Richards and F. G. Jackson. (*Proc. Amer. Acad.*, 1906, vol. 41, p. 21; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 261.)—The thermometer is standardized by means of solutions of known melting-points. For this purpose the thermometer is placed in a mixture of ice and water. The substance is then added and the thermometric reading obtained noted. Finally, the concentration of the substance added is determined analytically in the solution obtained, the real melting-point of a solution of this strength being then ascertained by reference to a table. In this way any number of points may be determined, and a curve of correction for the particular thermometer may be constructed. A. G. L.



A New Sodium Burner. (*Chem. Ztg.*, 1906, xxx., 835.)—The new burner, which is made by Carl Zéiss, consists essentially of a Bunsen burner provided with a nozzle (*C*), and a small salt tray, which can be adjusted at the edge of the flame by a screw (*J*). The tray consists of plate of pumice, 4 × 1 × ½ cm., which is soaked in a solution of salt before use. The light from the flame passes through a rectangular hole (*L*), 4 × 5 cm., cut in the screen (*K*), so as to yield a steady image of even intensity. A. G. L.

On a New Electrically-heated Microscope Stage. J. Zwintz and C. Thien. (*Zentralbl. Bakteriol.*, 1906, [1], vol. 42, p. 179; through *Chem. Ztg.*, 1906, xxx., *Rep.*, 341.)—The stage is heated electrically and provided with an automatic regulator. This consists practically of an air manometer, one limb of which is closed at one end and connected to a U-tube containing mercury at the other. At the bend of the U-tube a platinum contact-piece is sealed through the glass, whilst a graduated metallic rod is pushed to a certain depth into the open limb of the U-tube. If now the stage is heated to the desired temperature, the air in the closed limb of the manometer expands, pushing the mercury in the U-tube before it, and so making contact between the platinum contact-piece and the metallic rod. This closes the circuit of a battery which works a contact-breaker included in the heating circuit, and thus cuts off the latter. A. G. L.



Weighing Bottle for Liquids. K. Buschmann. (*Chem. Ztg.*, 1906, xxx., 1060.)—To use the weighing bottle shown in the figure, some of the liquid is poured into it, and the hollow stopper is inserted so that the hole (*e*) in the stopper is not opposite the groove (*a*) in the neck of the bottle. The bottle is then weighed, the stopper turned so as to bring (*e*) opposite (*a*), and some of the liquid is forced out by compressing the india-rubber ball on the stopper, which is then again turned into its first position, after which the bottle is again weighed. The bottle may be obtained from F. A. Kühnlenz, Frauenwalde i. Th., Germany. A. G. L.

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REVIEW.

Toxins and Antitoxins. By C. Oppenheimer and C. A. Mitchell. (London: C. Griffin and Company, Ltd. Crown 8vo.; cloth. Price 7s. 6d. net.)

Very few books of the size (250 pages) contain such a mass of detailed information upon any subject as has been crowded by the authors into this volume. There are innumerable references in the text and in the footnotes, and an excellent bibliography at the end. The book is difficult to read, but this is not so much the fault of the authors as of the subject. Since the existence of toxins and antitoxins was first surmised numerous workers have been engaged in isolating and studying them, and most complex theories have been evoked to explain their actions and interactions. At present we are threading an apparently most intricate maze, and much further exploration is necessary before we can say with certainty that the key to it has been found. The authors endeavour to simplify their labours by being very precise in their definitions. According to them a toxin is not a ptomaine or a toxalbumin, nor yet a proteid, but a complex body whose chemical structure is absolutely unknown, and which shows many remarkably close analogies with ferments. They have a specific physiological action, and each true toxin has its corresponding true antitoxin. Moreover, the authors appear to accept Ehrlich's theory in its entirety, and "rigorously exclude from the toxins any poisonous substance that is not a haptine." By this rigorous system of exclusion the number of bodies to be considered is reduced to a minimum. Section 1 is devoted to the True Toxins, Section 3 to the Vegetable Toxins (phytotoxins), and Section 4 to the Animal Toxins (zootoxins), whilst Section 2 deals with Endotoxins and other bacterial poisons.

To the bacteriologist and pathologist the work must be of the greatest value, but to the practical chemist or Public Analyst it can have but little interest. As there are no chemical tests whereby a toxin can be identified or even the presence of a toxin determined, their isolation can only be attempted by those who are licensed to experiment on animals.

Details are given here and there for the isolation of certain of the bodies under consideration, but no reference is made to poisons formed in articles of food or to food-poisoning. The very last paragraph, however, is interesting in the light of the recent revelations with reference to the slaughter of animals for canning purposes. Weichart has shown that the muscles of over-fatigued animals contain an apparently true toxin, which in large doses is fatal. It would appear, therefore, to be desirable that all animals should enjoy a period of rest before being slaughtered. J. C. T.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

An examination in biological chemistry was held on October 23 to 26, 1906. Two Associates who were examined for the Certificate passed: Clarke, Robert William, A.I.C.; Filmer, Reginald Mead, B.Sc. (Lond.), A.I.C. Two candidates examined for the Associateship passed: Gimingham, Conrad Theodore, University College, London; Whatmough, Wilfred Ambrose, King's College, London, and under A. H. M. Muter, F.I.C. The examiner in biological chemistry was Arthur Harden, Esq., D.Sc. (Vict.), Ph.D. (Erlangen), F.I.C. The examiners in chemistry were Walter William Fisher, Esq., M.A. (Oxon), F.I.C., and Professor George Gerald Henderson, M.A., D.Sc. (Glasgow), F.I.C.

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